

# **Interim Report on Task 1.4: Impurity Effects Part 2 of 2 Appendices To Lawrence Livermore National Laboratory for Contract B345772**

M. W. A. Stewart, E. R. Vance, and R. A. Day

***U.S. Department of Energy***

Lawrence  
Livermore  
National  
Laboratory

**February 26, 1999**

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.

This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.



## **Interim Report on Task 1.4: Impurity Effects**

**Part 2 of 2**

### **APPENDICES**

**To Lawrence Livermore National Laboratory for Contract  
B345772**

M W A Stewart, E R Vance and R A Day

26 February 1999

R99m011

# **Interim Report on Task 1.4: Impurity Effects**

## **Appendices**

### **To Lawrence Livermore National Laboratory for Contract B345772**

DATE ISSUED

26 February 1999

ISSUED TO

Lawrence Livermore National Laboratories

REPORT NUMBER

R99m011

JOB NUMBER

713m

AUTHORS

Stewart, Vance and Day

APPROVED BY

E R Vance

Australian Nuclear Science and Technology Organisation  
Postal Address: Private Mail Bag 1, Menai, NSW 2234, Australia  
Materials Division: Telephone +61 2 9717 3265 • Facsimile +61 2 9543 7179

---

# TABLE OF CONTENTS

APPENDIX A - SCANNING ELECTRON MICROGRAPHS OF THE Pu/U-DOPED, Ce/U-DOPED AND Th/Np-DOPED SAMPLES FIRED IN ARGON AT 1350°C FOR 4 HOURS

APPENDIX B - SCANNING ELECTRON MICROGRAPHS OF THE Pu/U-DOPED AND Ce/U-DOPED SAMPLES FIRED IN AIR AT 1350°C FOR 4 HOURS

APPENDIX C - SCANNING ELECTRON MICROGRAPHS OF THE Pu/U-DOPED AND Ce/U-DOPED SAMPLES FIRED IN 3.7 % H<sub>2</sub>/ARGON AT 1350°C FOR 4 HOURS

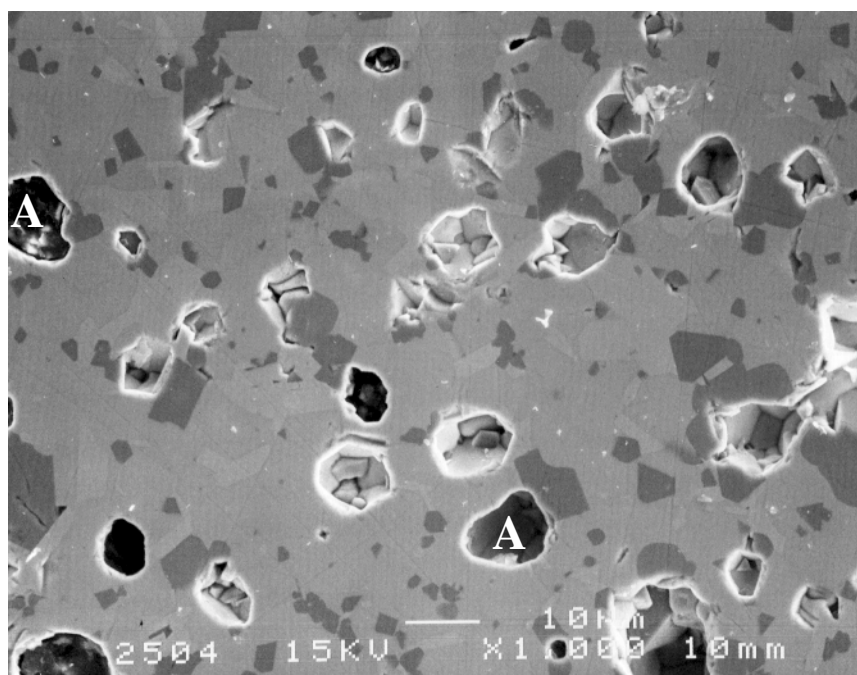
APPENDIX D - SCANNING ELECTRON MICROGRAPHS OF THE LLNL GLASS-DOPED SAMPLES

APPENDIX E - X-RAY DIFFRACTION RESULTS OF THE SAMPLES FROM TASK 1.4

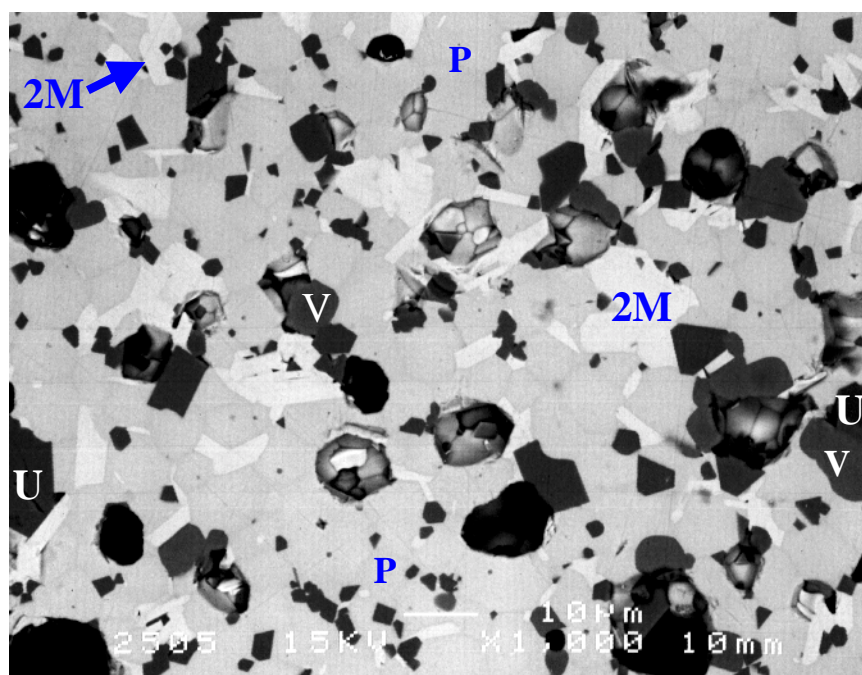
APPENDIX F - ENERGY DISPERSIVE X-RAY SPECTROSCOPY RESULTS ON THE SCANNING ELECTRON MICROSCOPY SAMPLES SHOWN IN APPENDICES A TO D

## **APPENDIX A**

**SCANNING ELECTRON MICROGRAPHS OF  
THE Pu/U-DOPED, Ce/U-DOPED AND Th/Np-  
DOPED SAMPLES FIRED IN ARGON AT 1350°C  
FOR 4 HOURS**



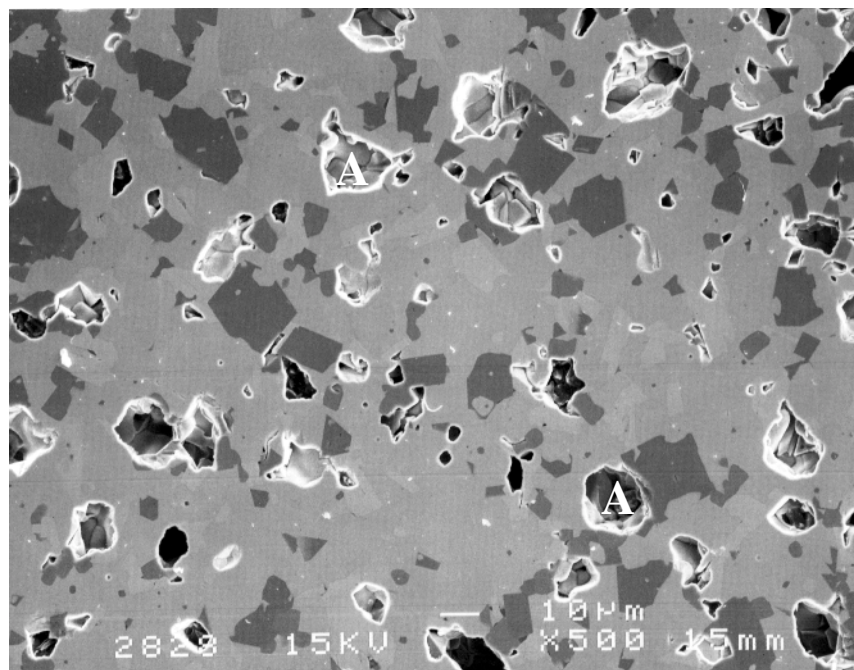
(a)



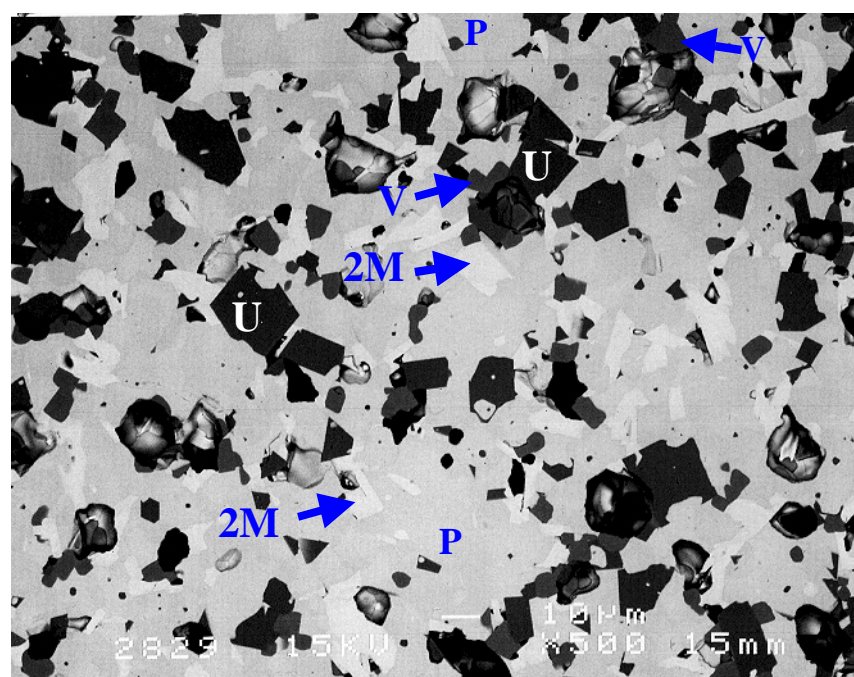
(b)

— 10 μm

Figure A-1: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980088 (Task 1.4, 2+ (Mg, Co, Ni, Cu, Zn)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a matrix of pyrochlore (P), 2M zirconolite (2M, light grey-white), perovskite (V, dark-grey roundish grains), ulvospinel (U, darkest-grey angular grains) and a small amount of porosity (A).



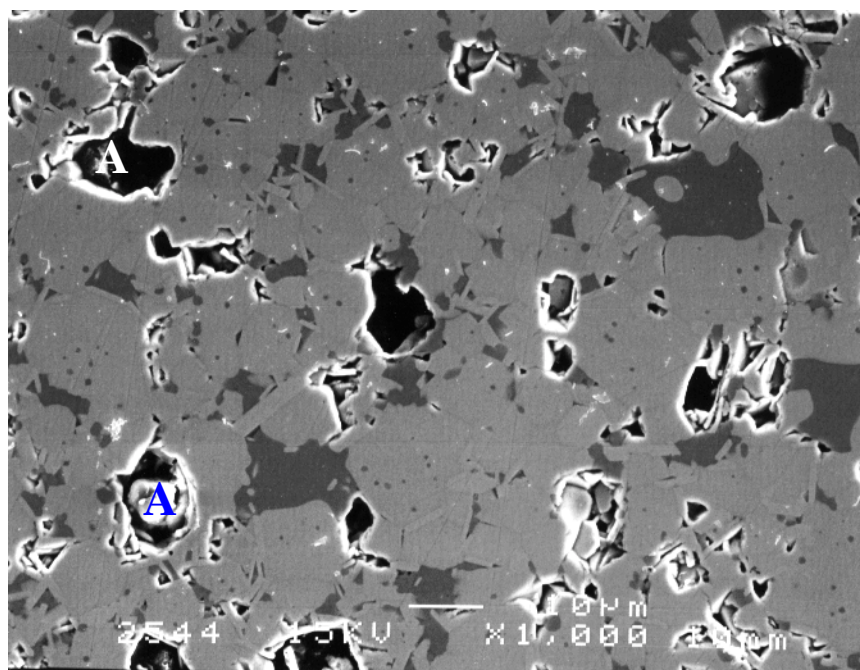
(a)



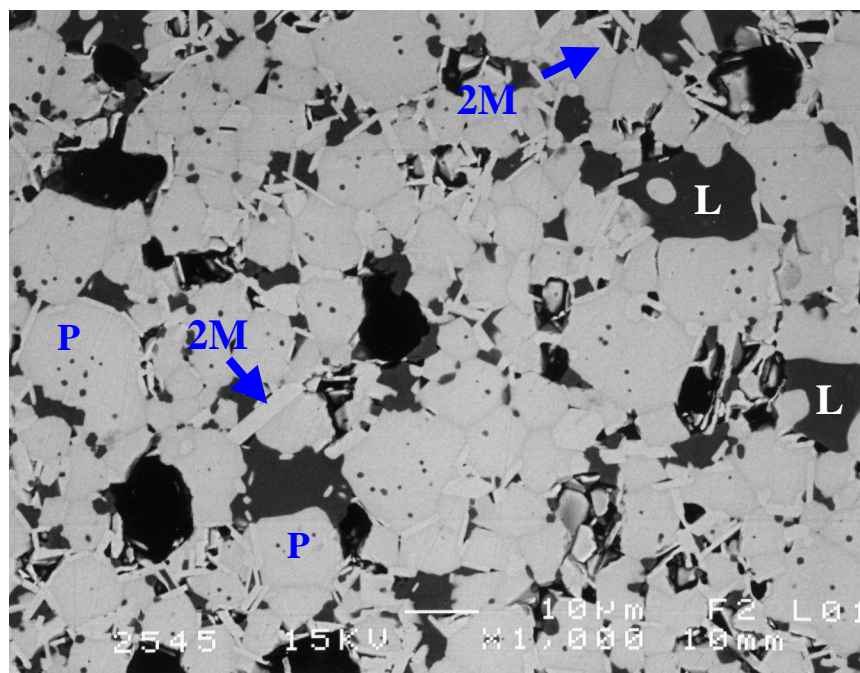
(b)

— 10 μm

Figure A-2: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980224 (Task 1.4, 2+ (Mg, Co, Ni, Cu, Zn, Mn, Fe)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a matrix of pyrochlore (P), 2M zirconolite (2M, lightest grey), perovskite (V, dark-grey grains), ulvospinel (U, darkest-grey angular grains) and porosity (A).



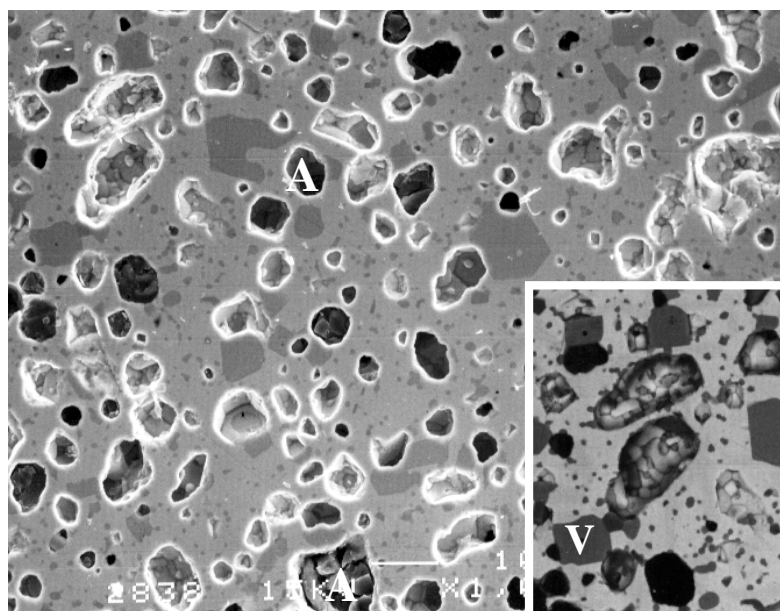
(a)



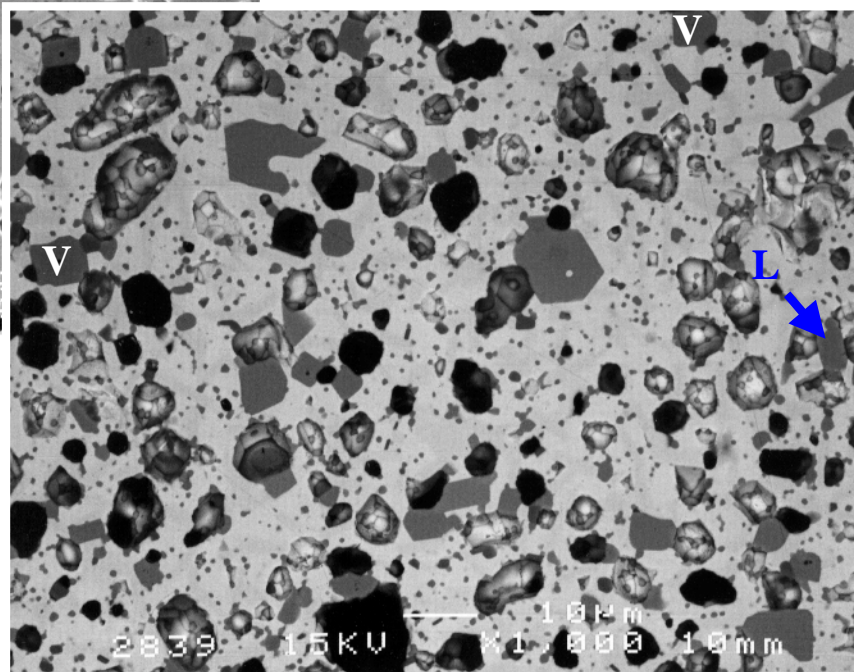
(b)

— 10 μm

Figure A-3: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980090 (Task 1.4, 3+ (Al, V, Cr, Fe, Mn, Ga)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a pyrochlore (P), 2M zirconolite (2M, lighter elongated grains), a lovingite-like phase (L, dark-grey roundish grains), and porosity (A).

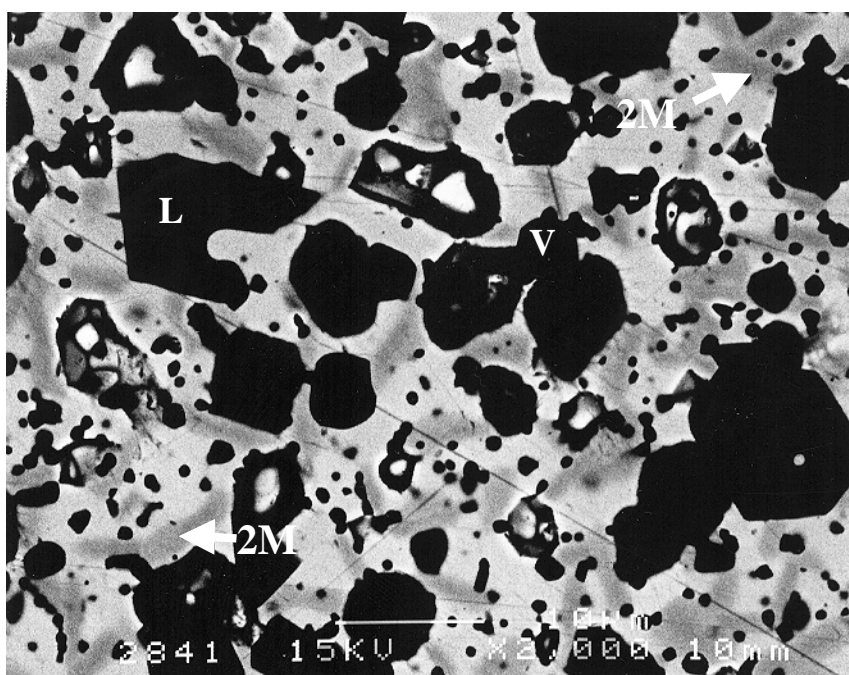


(a)



(b)

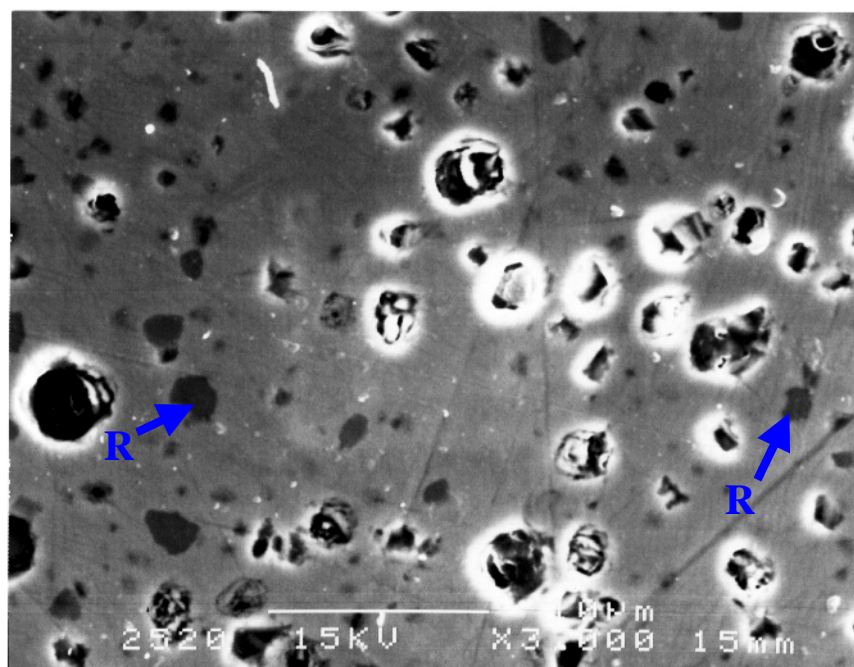
— 10  $\mu\text{m}$



(c)

— 10  $\mu\text{m}$

Figure A-4: (a) Secondary electron micrograph and (b) and (c) backscattered electron micrograph of mws980225 (Task 1.4, 3+ (Al, Cr, Fe, Mn, Ga)-doped batch, sintered at 1350°C in Ar for 4 hours). Figure (c) is taken at a different contrast to illustrate the 2M zirconolite. The pellet consists of a matrix of pyrochlore and 2M zirconolite (2M, elongated-platy grains in the matrix (c)), a loweringite-like phase (L, dark-grey).



(a)

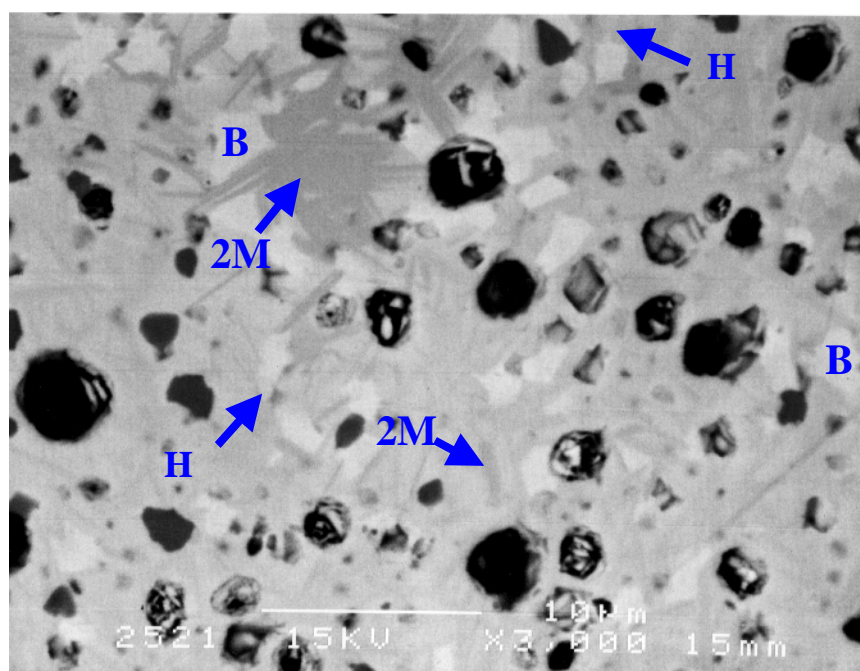
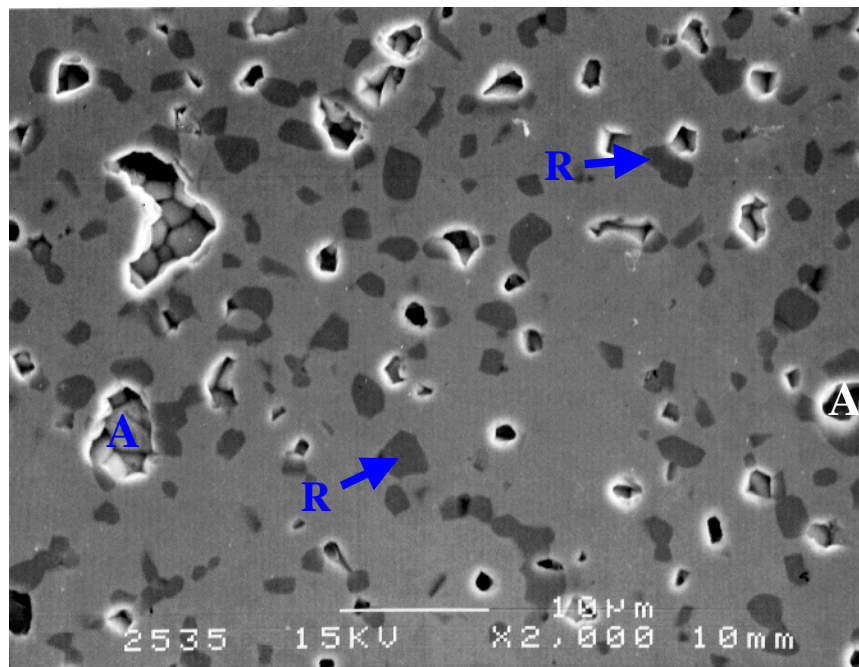
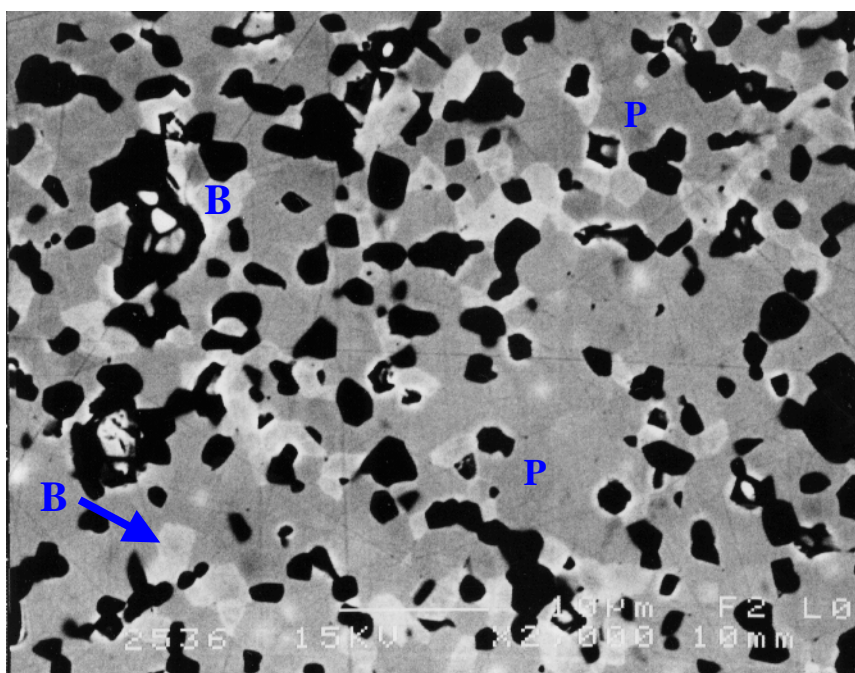
(b)  10  $\mu\text{m}$ 

Figure A-5: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980092 (Task 1.4, 4+ (Zr, Sn, Hf)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a matrix, which is a mixture of pyrochlore and 4M zirconolite, 2M zirconolite (2M, darker grey elongated grains), Hf-Zr-titanate (H, small, light-grey roundish grains), brannerite, (B, white grains), Hf-doped rutile (R, black to dark-grey grains), and porosity (A).



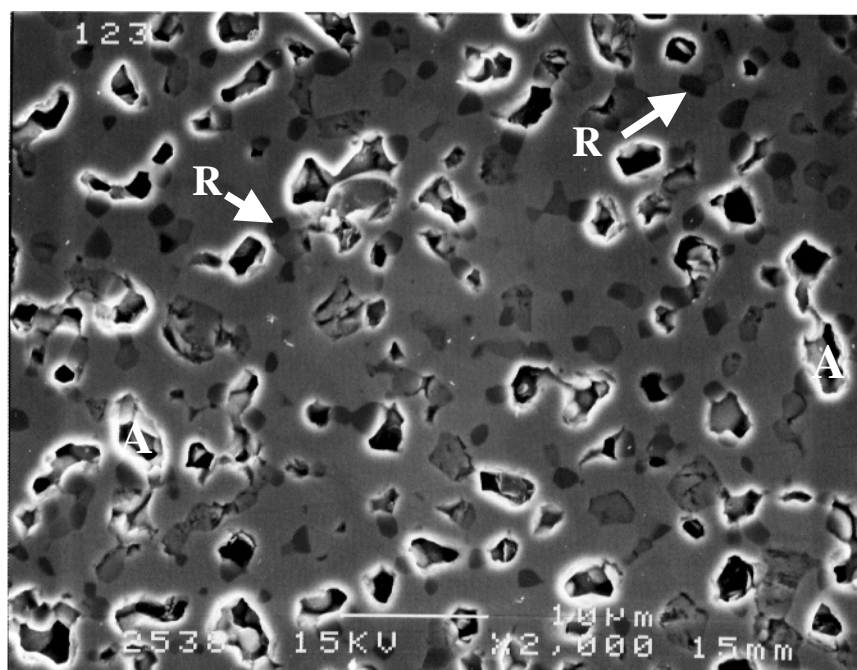
(a)



(b)

10 μm

Figure A-6: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980094 (Task 1.4, 5+ (Nb, Ta)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a pyrochlore matrix (P), brannerite (B, light-grey grains), rutile (R, dark-grey grains), and porosity (A).



(a)

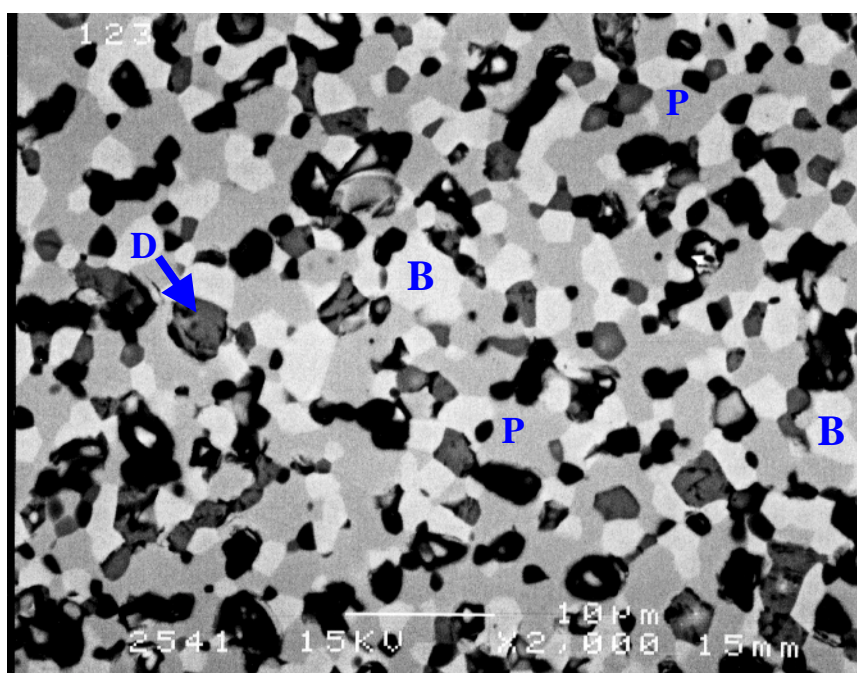
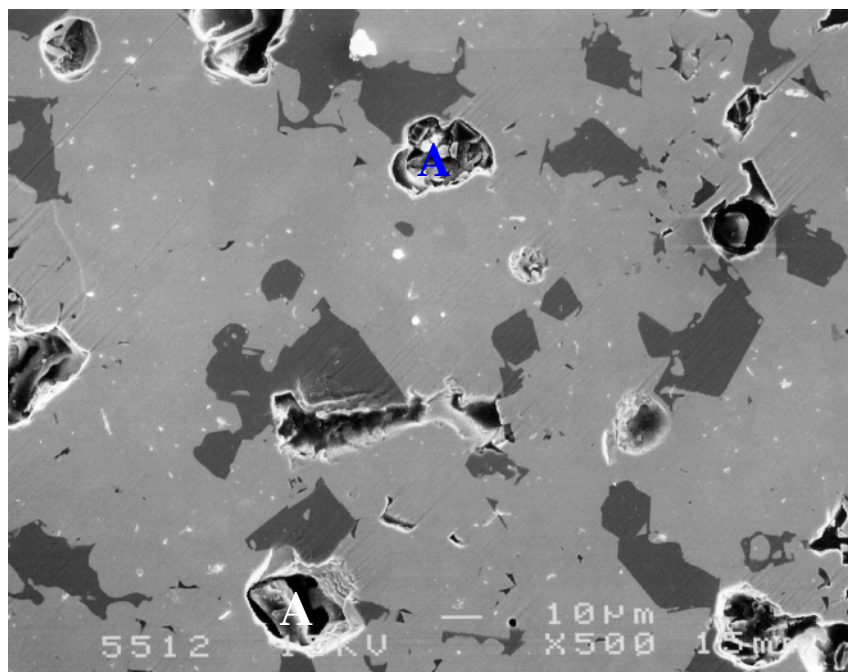
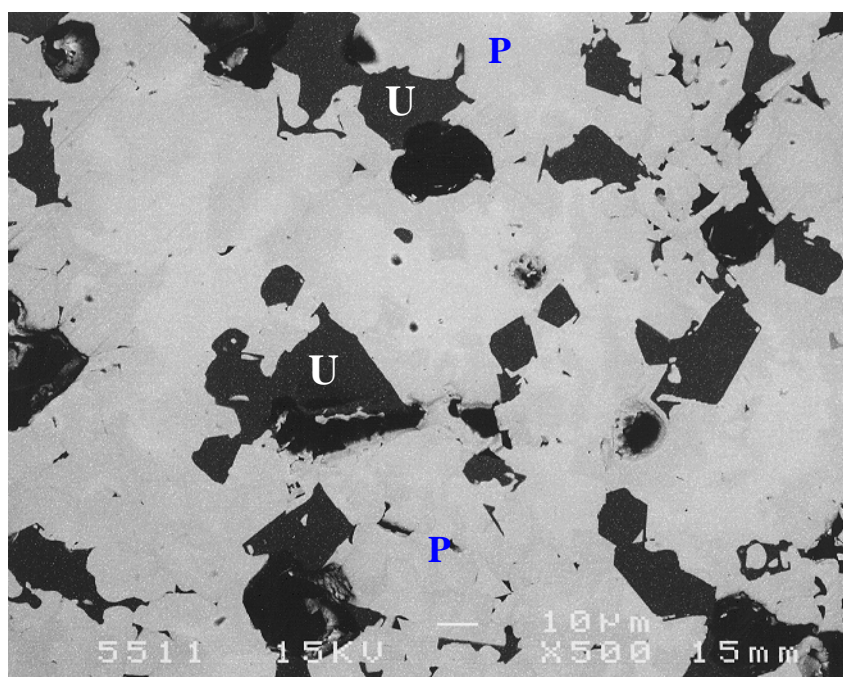
(b)  10  $\mu\text{m}$ 

Figure A-7: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980096 (Task 1.4, 6+ (Mo, W)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a pyrochlore matrix (P), brannerite (B, light-grey grains), rutile (R, dark-grey grains), and powellite/scheelite ( $\text{Ca}(\text{Mo}, \text{W})\text{O}_4$ ) (D, mid-grey phase with some associated porosity) and porosity (A).



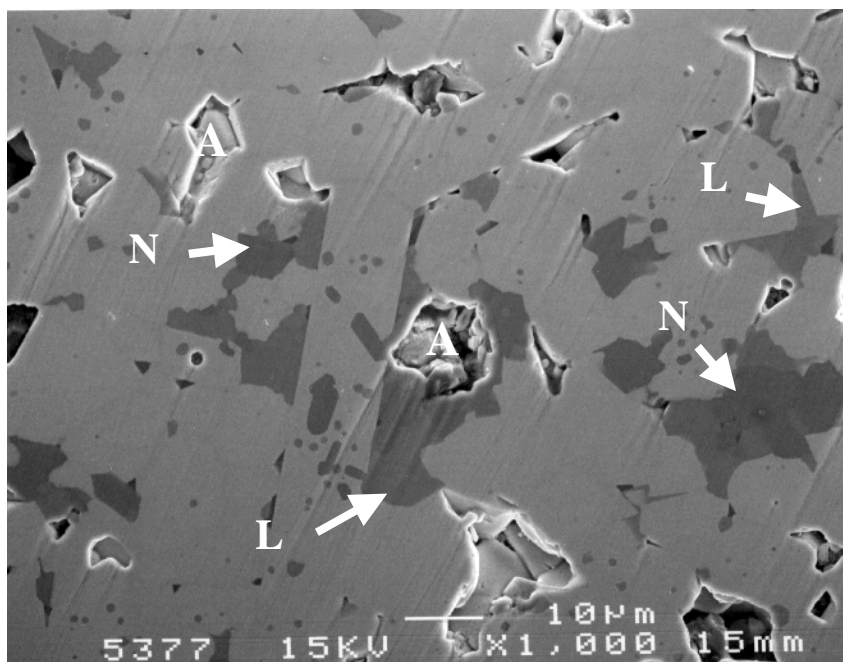
(a)



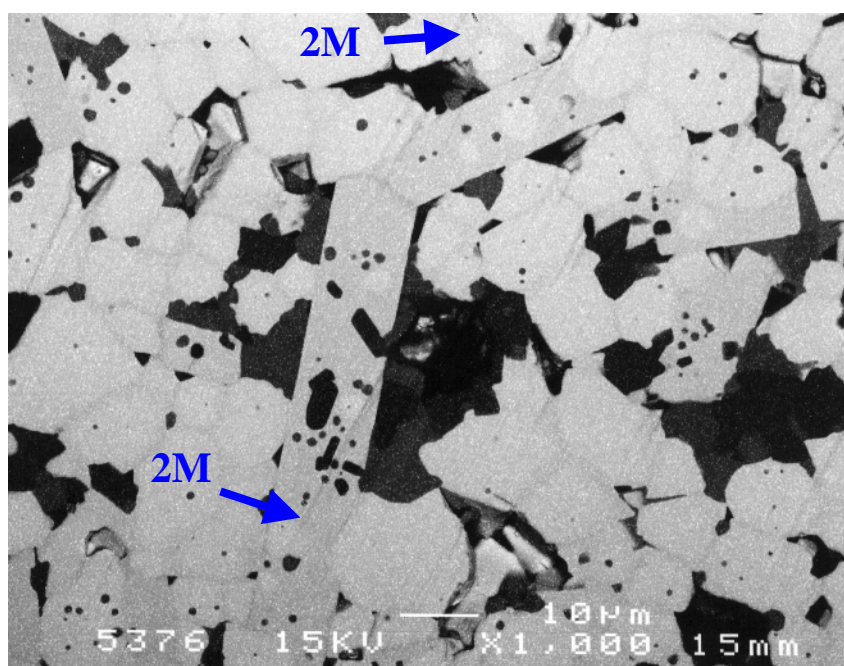
(b)

— 10 μm

Figure A-8: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu107-01 (Task 1.4, 2+ (Mg, Co, Ni, Cu, Zn, Mn, Fe) doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a matrix of pyrochlore of two distinct compositions, bright and dark domains (P), ulvöspinel (U, dark-grey grains) and porosity (A).



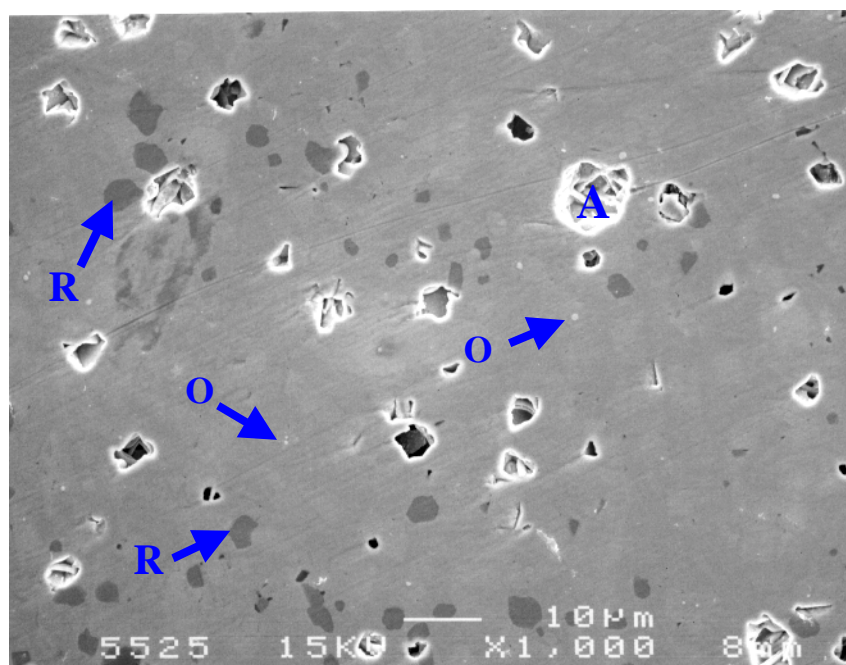
(a)



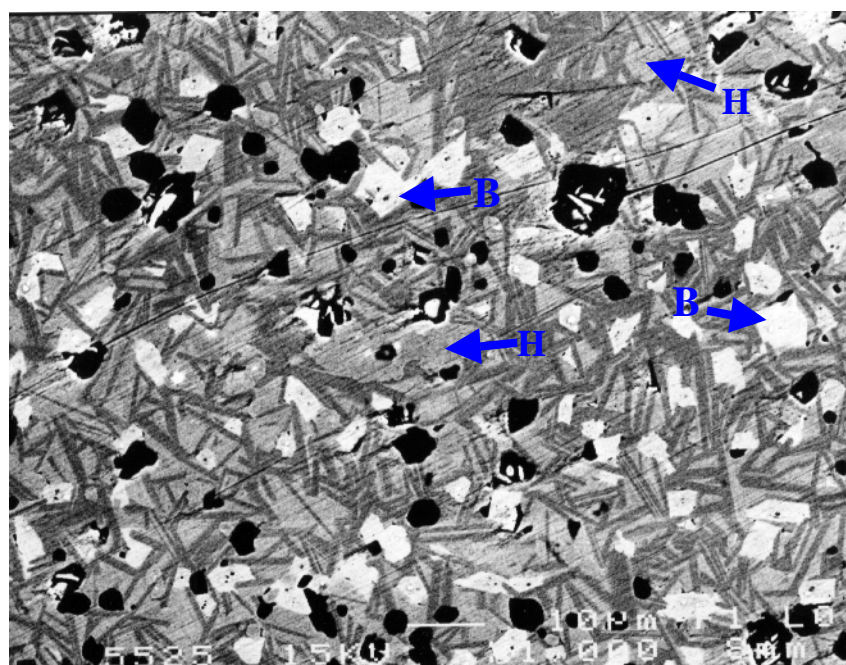
(b)

— 10 μm

Figure A-9: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu108-01 (Task 1.4, 3+ (Al, Cr, Fe, Mn, Ga)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a matrix of pyrochlore (P, light grey) and 2M zirconolite (2M), lovingite (L, dark-grey), magnetoplumbite-like phase (N, black in (b)) and porosity (A).



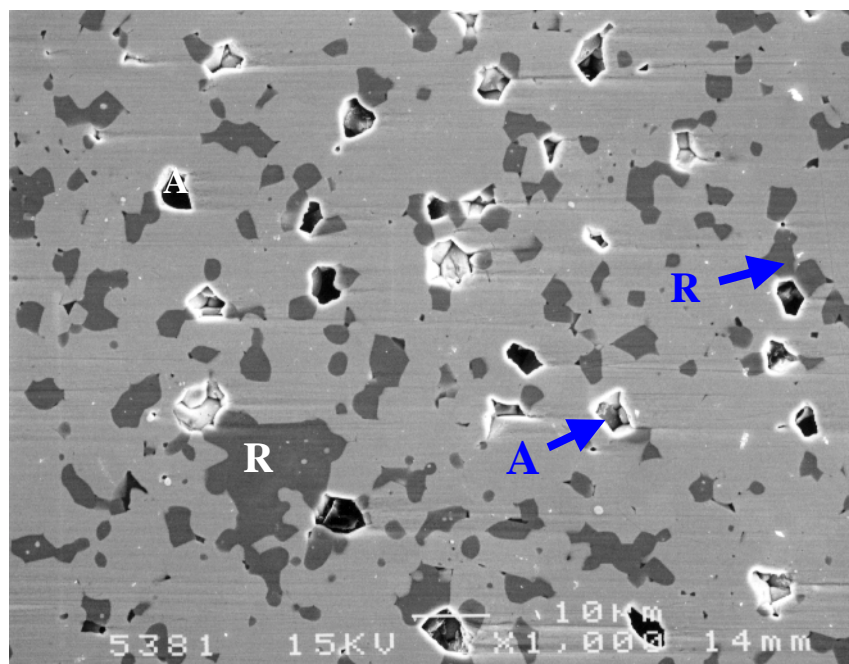
(a)



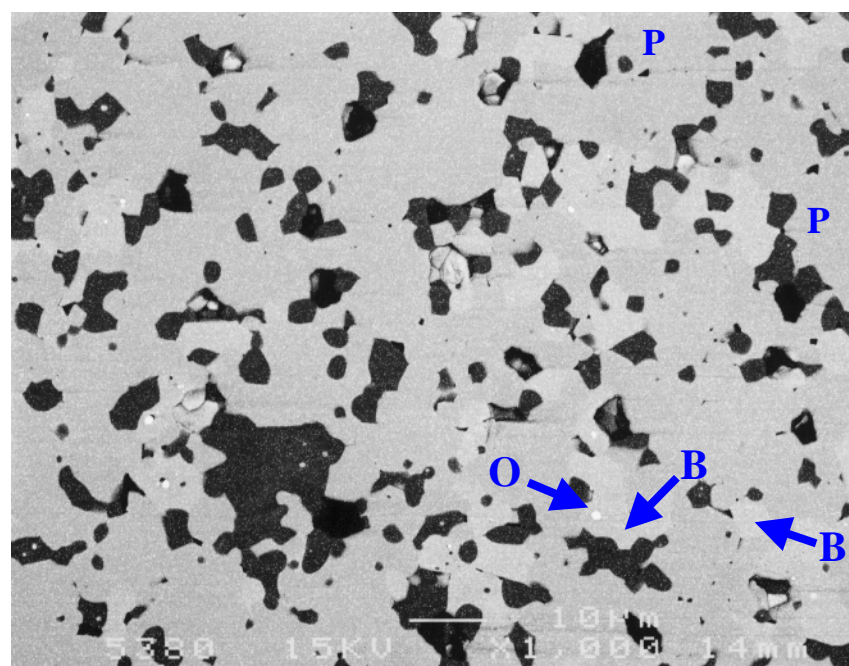
(b)

— 10 μm

Figure A-10: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu109-01 (Task 1.4, 4+ (Zr, Sn, Hf)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a matrix, which is a mixture of pyrochlore (grey) and 2M zirconolite (dark-grey, elongated grains in (b)). Also present are Hf-Zr-titanate (H), brannerite, (B, lightest-grey grains), Hf-doped rutile (R, Black to dark-grey grains), and porosity (A). There is also < 1 vol. % (Pu,U)O<sub>2</sub> (O, white spots inside brannerite grains).



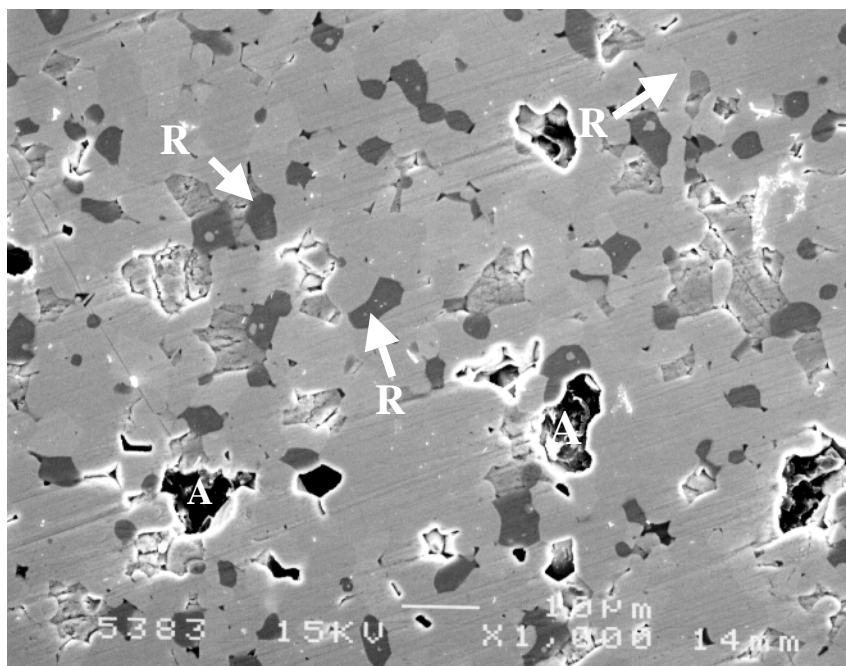
(a)



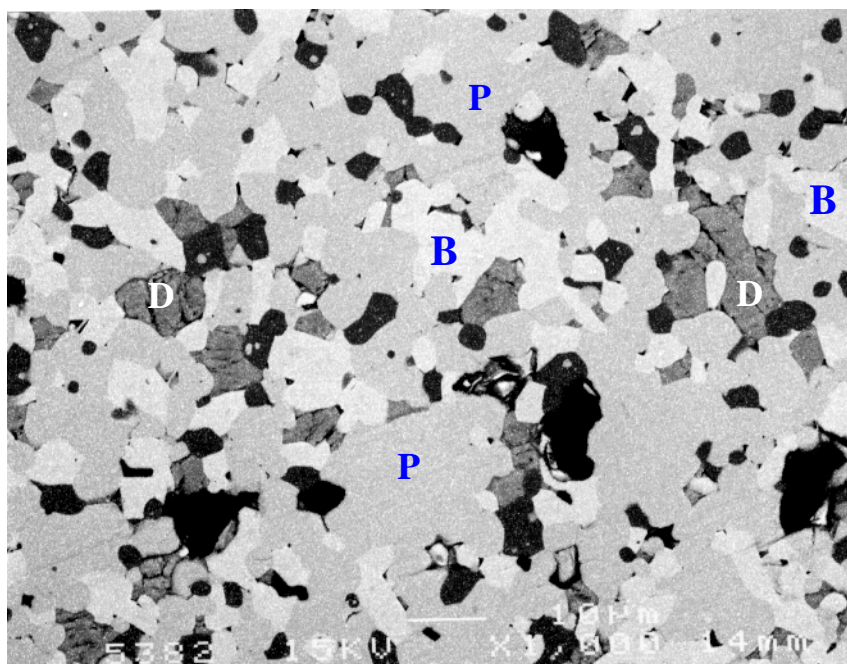
(b)

— 10 μm

Figure A-11: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu110-01 (Task 1.4, 5+ (Nb, Ta)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a pyrochlore matrix (P), brannerite (B, lighter grey than pyrochlore), rutile (R, dark-grey grains) << 1 vol. % (Pu,U)O<sub>2</sub> (O, white spots in brannerite), and porosity (A).



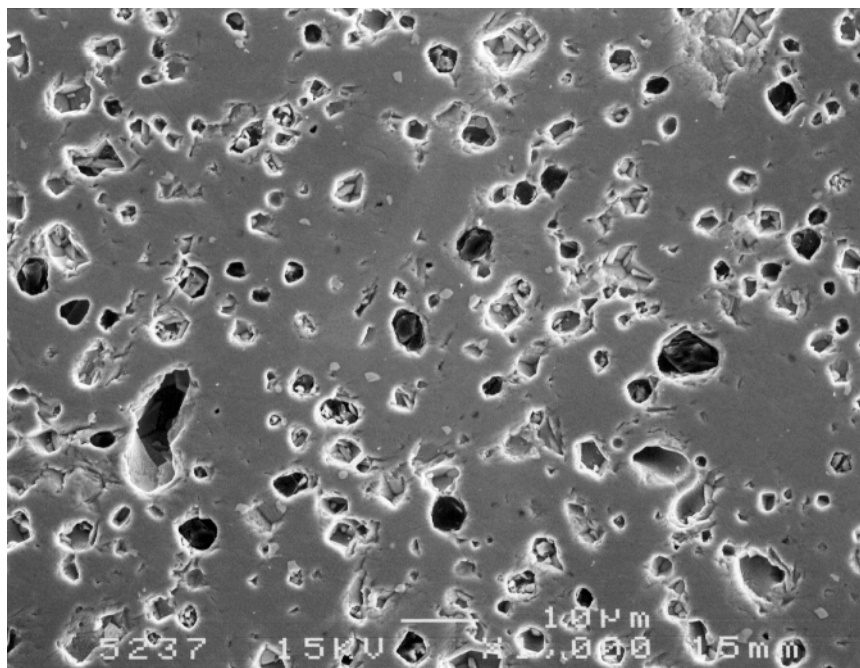
(a)



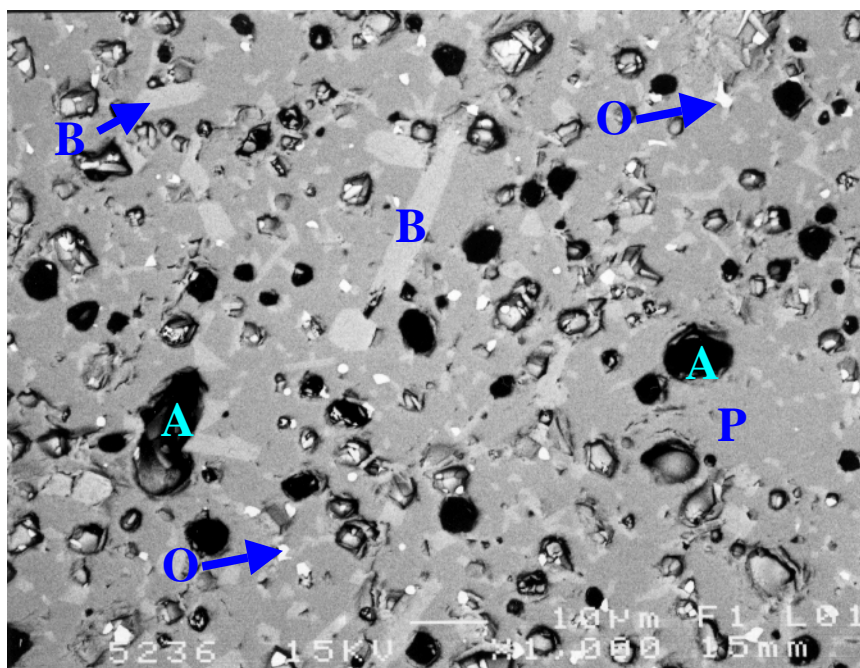
(b)

———— 10 μm

Figure A-12: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu111-01 (Task 1.4, 6+ (Mo, W)-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of a pyrochlore matrix (P), brannerite (B, light-grey grains), rutile (R, dark-grey grains), and powellite/scheelite ( $\text{Ca}(\text{Mo}, \text{W})\text{O}_4$ ) (D, mid-grey phase, which has some associated porosity) and porosity (A).



(a)



(b)

Figure A-13: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Np48 (Task 1.4, Np/Th-doped batch, alkoxide-route, sintered at 1350°C in Ar for 4 hours). The pellet consists of pyrochlore matrix (P) with Th/Np/Ce/U-brannerite (B, light-grey) grains,  $(\text{Ce,Gd,Th,U,Np})\text{O}_2$  (O, fine white grains) and porosity (A).

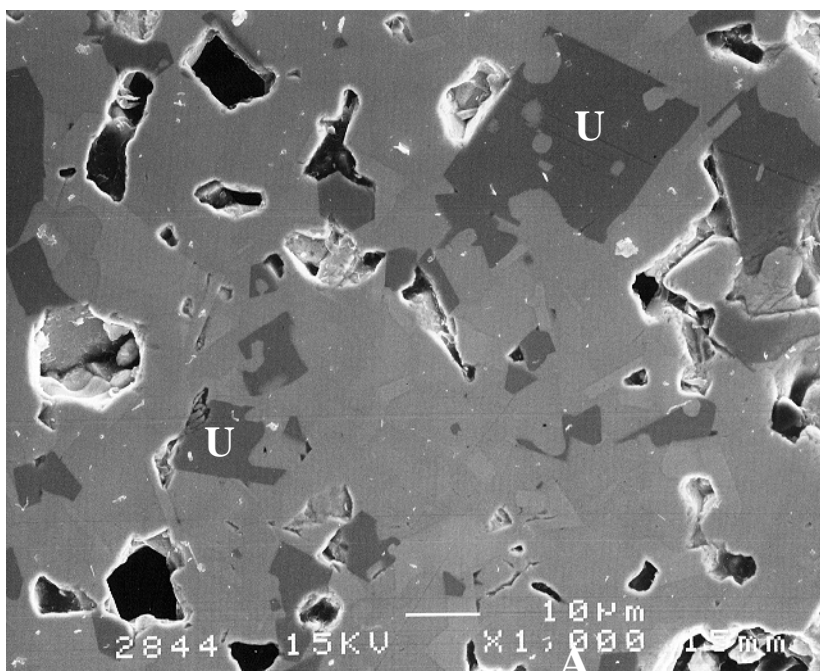
## **APPENDIX B**

### **SCANNING ELECTRON MICROGRAPHS OF THE Pu/U-DOPED AND Ce/U-DOPED SAMPLES FIRED IN AIR AT 1350°C FOR 4 HOURS**

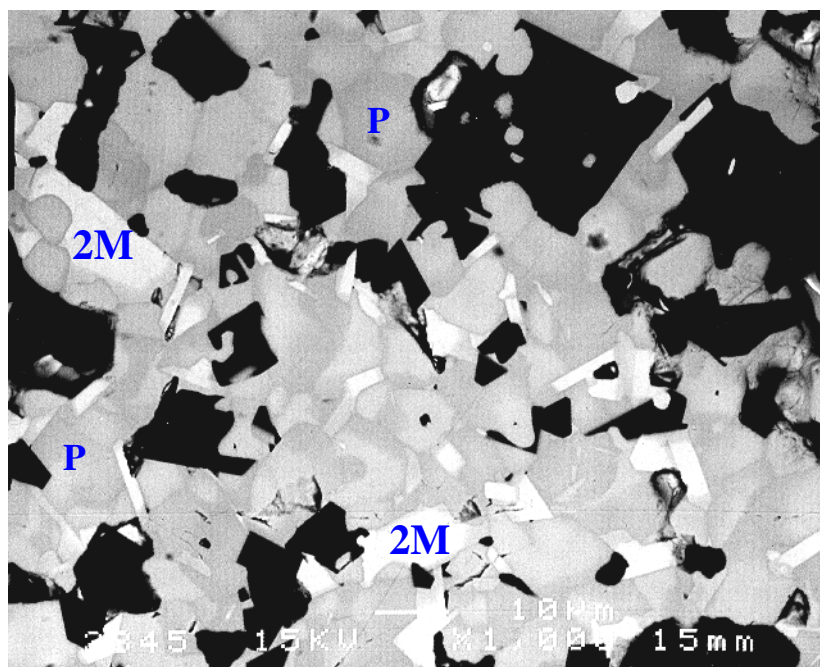
(a)



(b)

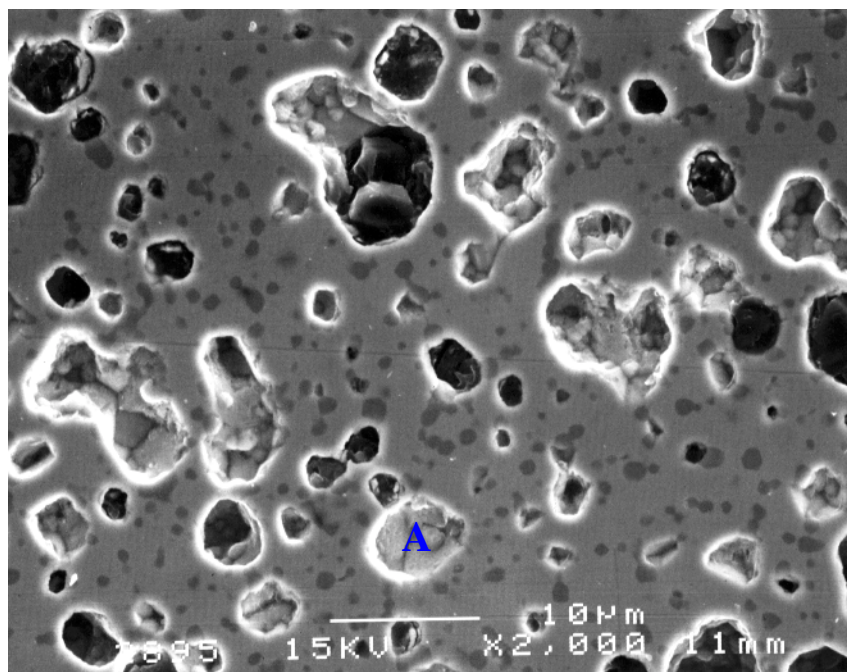


(c)

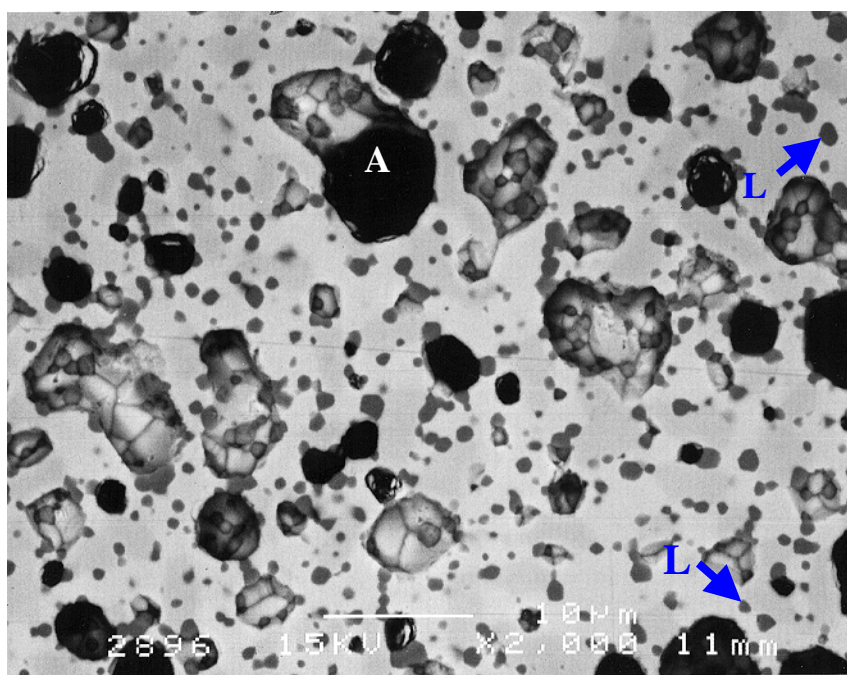


10  $\mu\text{m}$

Figure B-1: (a) Secondary electron micrograph and (b) backscattered electron micrograph of



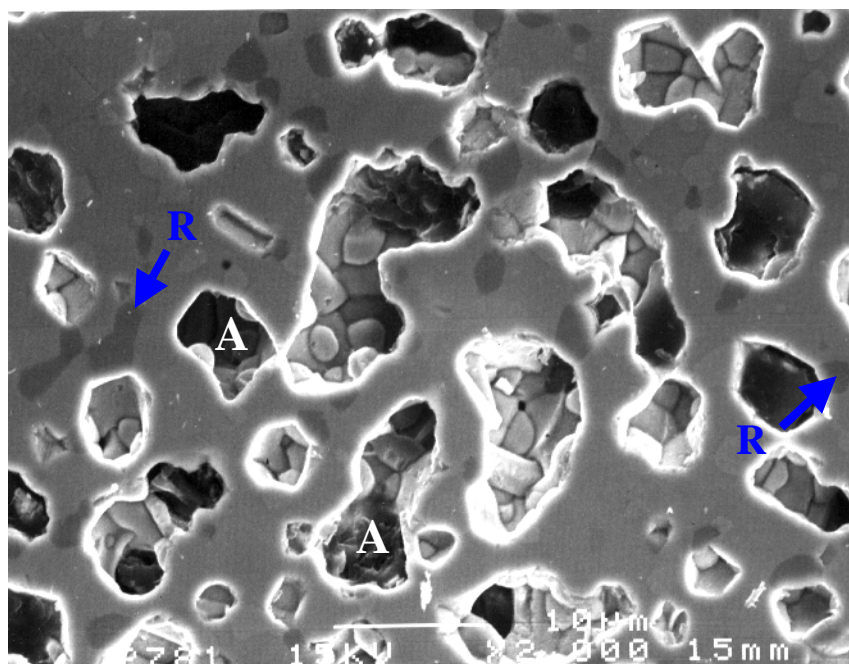
(a)



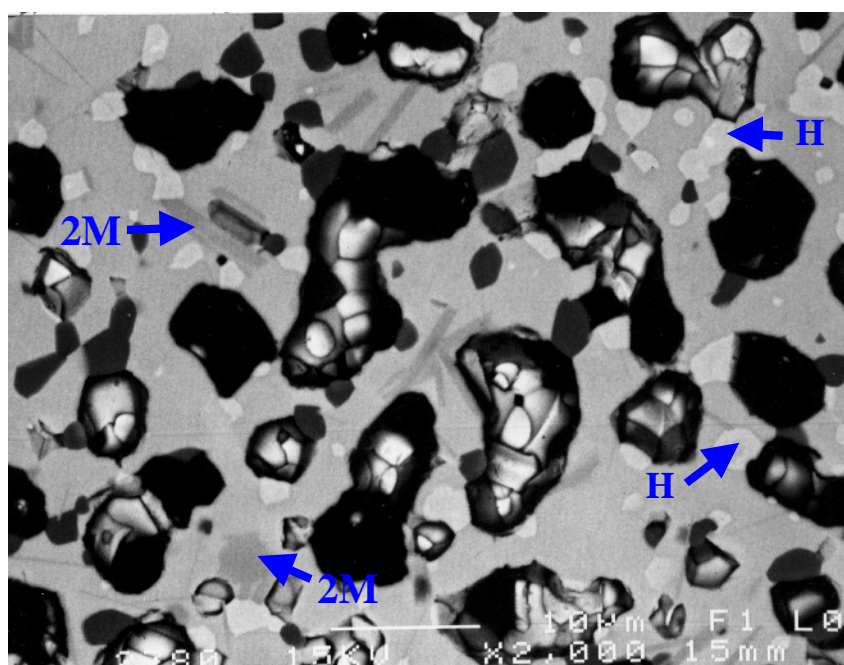
(b)

10 μm

Figure B-2: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980227 (Task 1.4, 3+ (Al, Cr, Fe, Mn, Ga)-doped batch, sintered at 1350°C in air for 4 hours). The pellet consists of a matrix of pyrochlore (lighter grey) and 2M zirconolite (darker-grey), a loweringite-like phase (L, fine dark-grey grains), and porosity (A).



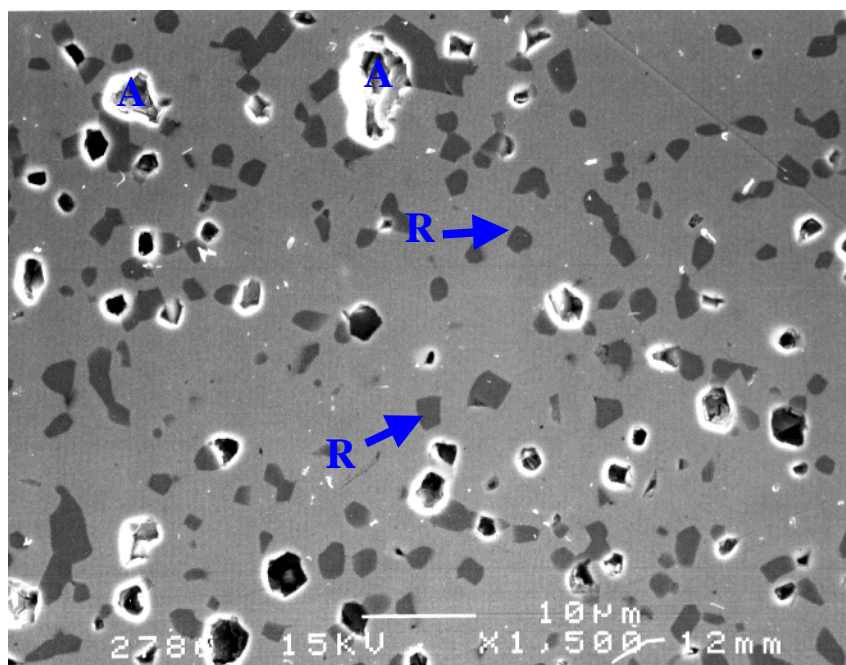
(a)



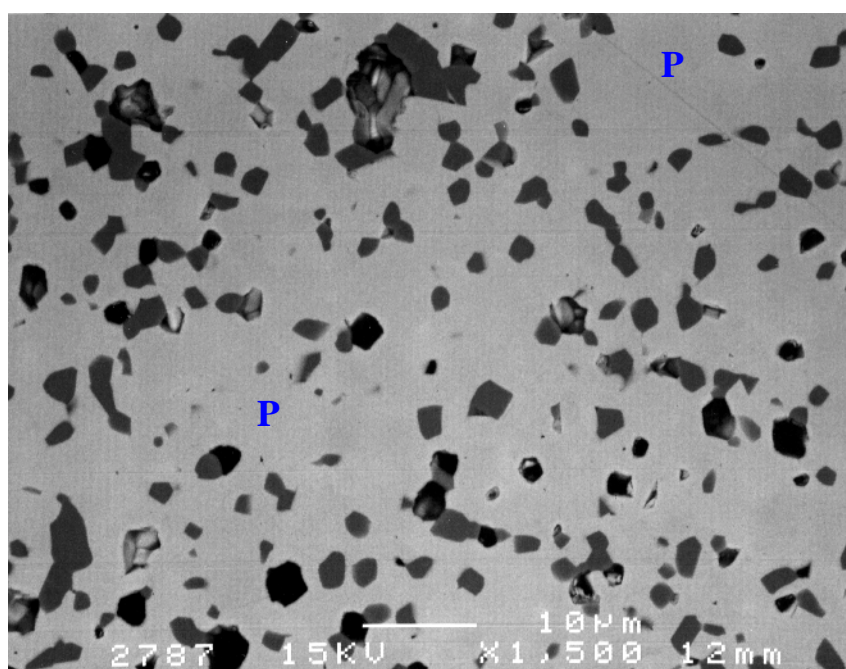
(b)

10 μm

Figure B-3: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980201 (Task 1.4, 4+ (Zr, Sn, Hf)-doped batch, sintered at 1350°C in air for 4 hours). The pellet consists of a pyrochlore matrix (P); 2M zirconolite (2M, darker grey elongated grains), Hf-Zr-titanate (H, small, light-grey roundish grains), Hf-doped rutile (R, Black to dark-grey grains), and porosity (A).



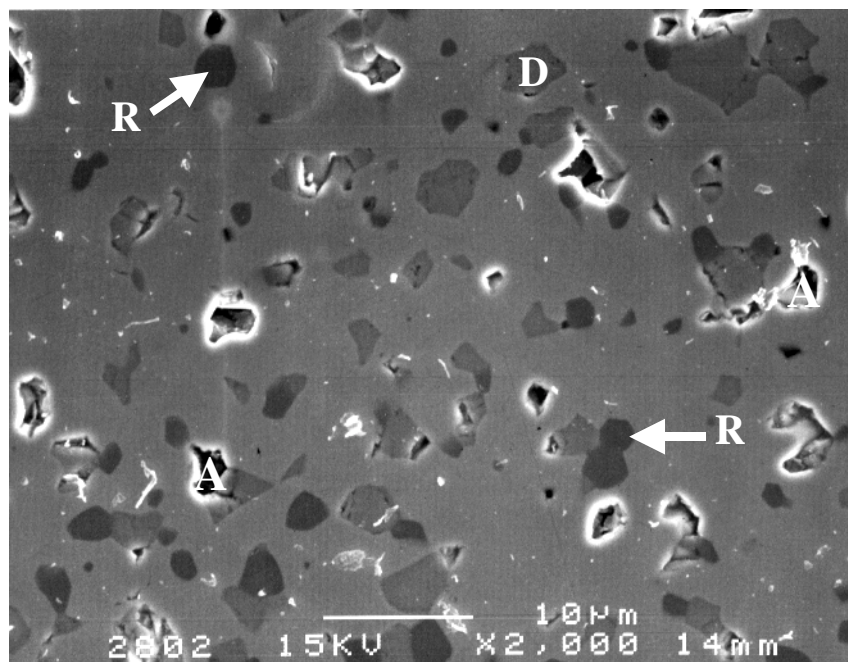
(a)



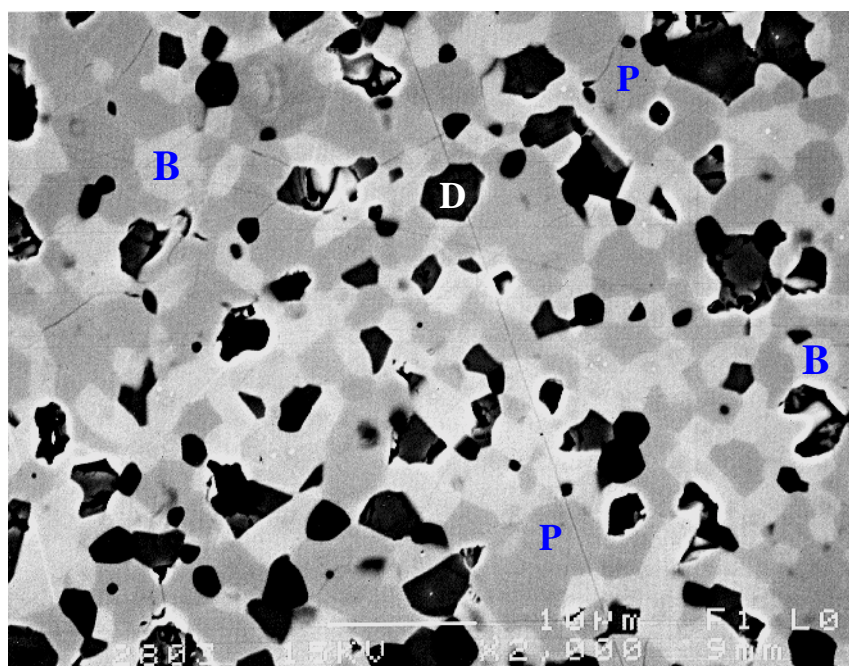
(b)

— 10 μm

Figure B-4: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980202 (Task 1.4, 5+ (Nb, Ta)-doped batch, sintered at 1350°C in air for 4 hours). The pellet consists of a pyrochlore matrix (P), rutile (R, dark-grey grains), and porosity (A).



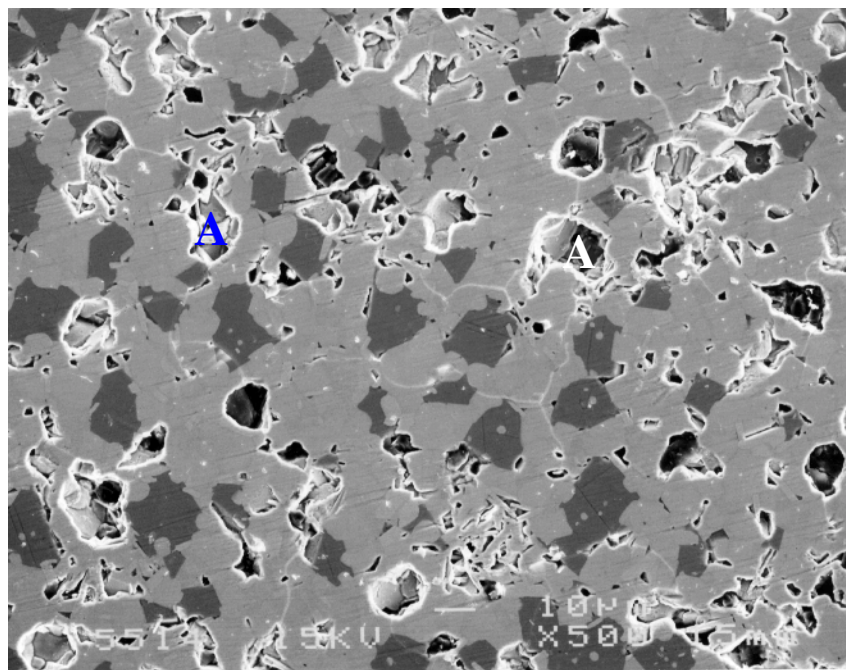
(a)



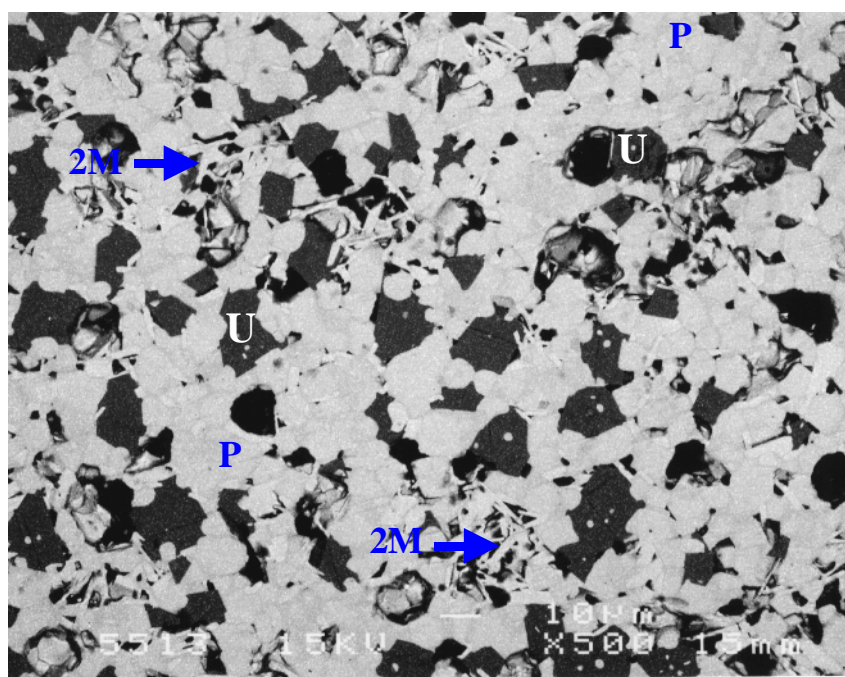
(b)

10 μm

Figure B-5: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980203 (Task 1.4, 6+ (Mo, W)-doped batch, sintered at 1350°C in air for 4 hours). The pellet consists of a pyrochlore matrix (P), brannerite (B, light-grey grains), rutile (R, dark-grey grains), and powellite/scheelite ( $\text{Ca}(\text{Mo}, \text{W})\text{O}_4$ ) (D, mid-grey phase with some associated porosity) and porosity (A).



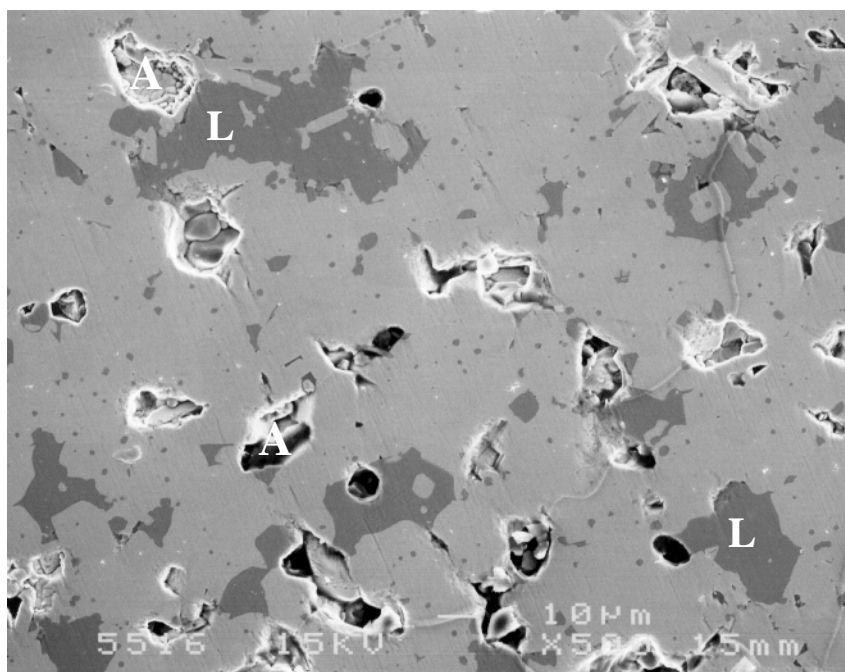
(a)



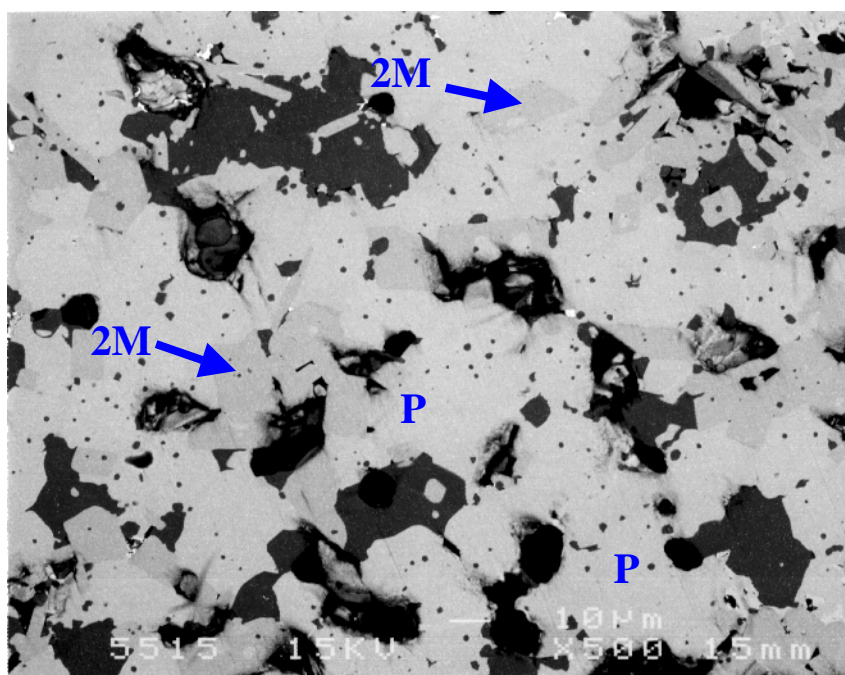
(b)

— 10 µm

Figure B-6: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu107-02 (Task 1.4, 2+ (Mg, Co, Ni, Cu, Zn, Mn, Fe) doped batch, sintered at 1350°C in air for 4 hours). The pellet consists of a matrix of pyrochlore (P), 2M zirconolite (2M, elongated-plate-like grains), ulvöspinel (U, dark-grey grains) and porosity (A).



(a)

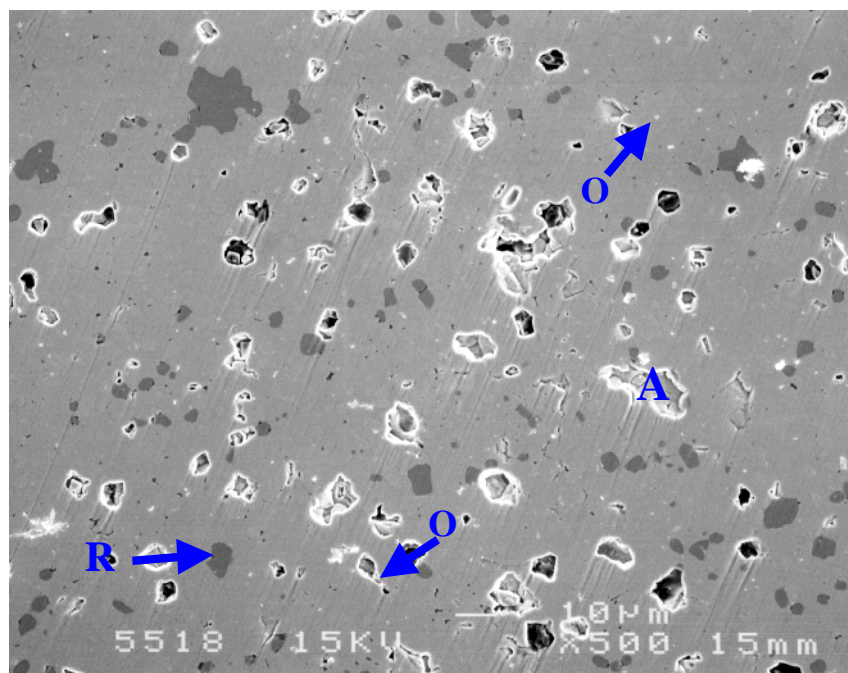


(b)

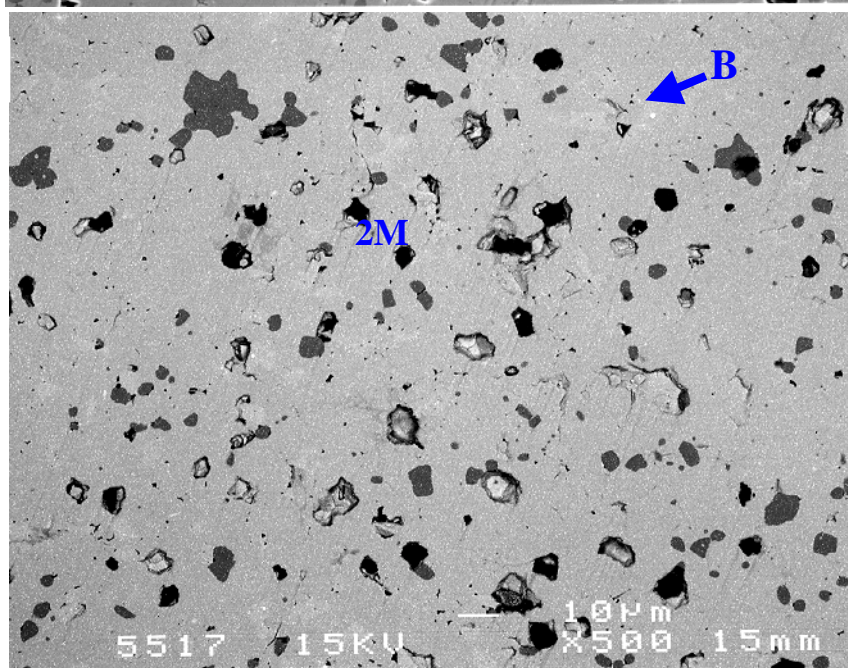
— 10 µm

Figure B-7: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu108-02 (Task 1.4, 3+ (Al, Cr, Fe, Mn, Ga)-doped batch, sintered at 1350°C in air for 4 hours). The pellet consists of a matrix of pyrochlore (P, light grey) and 2M zirconolite (2M, darker grey in matrix), a lovingite-like phase (L, dark-grey), << 1 vol. % (Pu,U)O<sub>2</sub> (O, white spots) and porosity (A).

(a)



(b)



— 10 μm

(c)

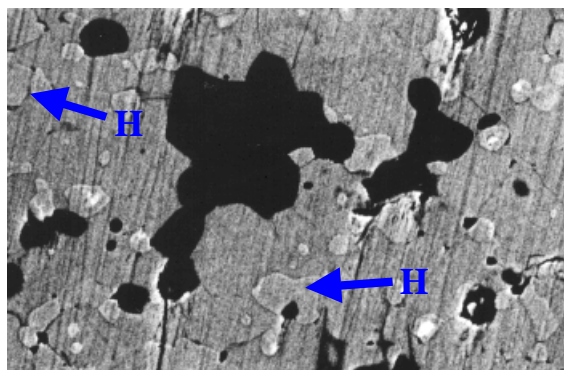
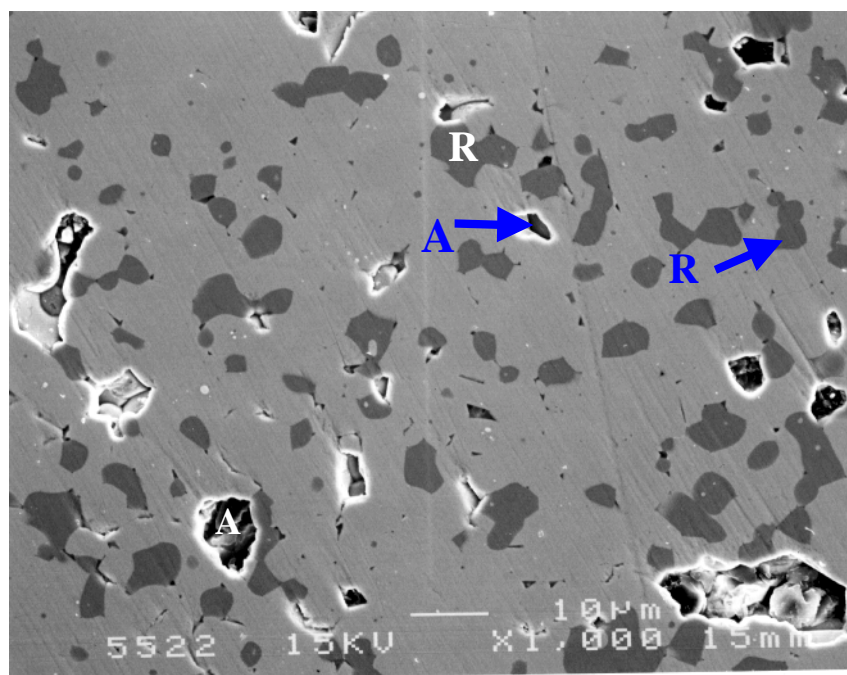
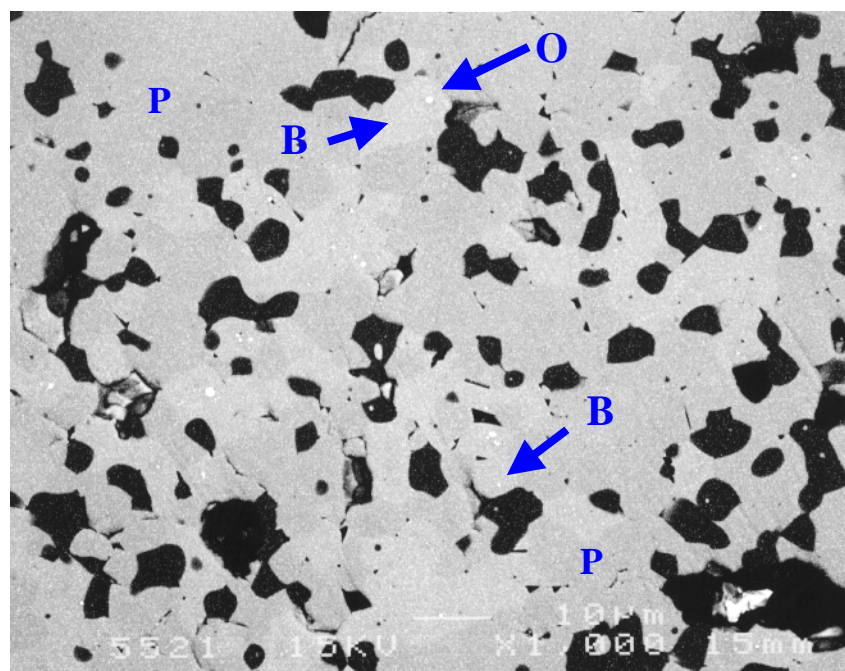


Figure B-8: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu109-01 (Task 1.4, 4+ (Zr, Sn, Hf)-doped batch, sintered at 1350°C in air for 4 hours). (c) is a blow up of part of (b) with the contrast varied to illustrate the Hf-Zr-titanate phase. The pellet consists of a matrix of pyrochlore (grey), 2M zirconolite (dark-grey grains in (b)). Also present are Hf-Zr-titanate (H) which has a similar contrast to the pyrochlore (fig. (c) is given to illustrate this



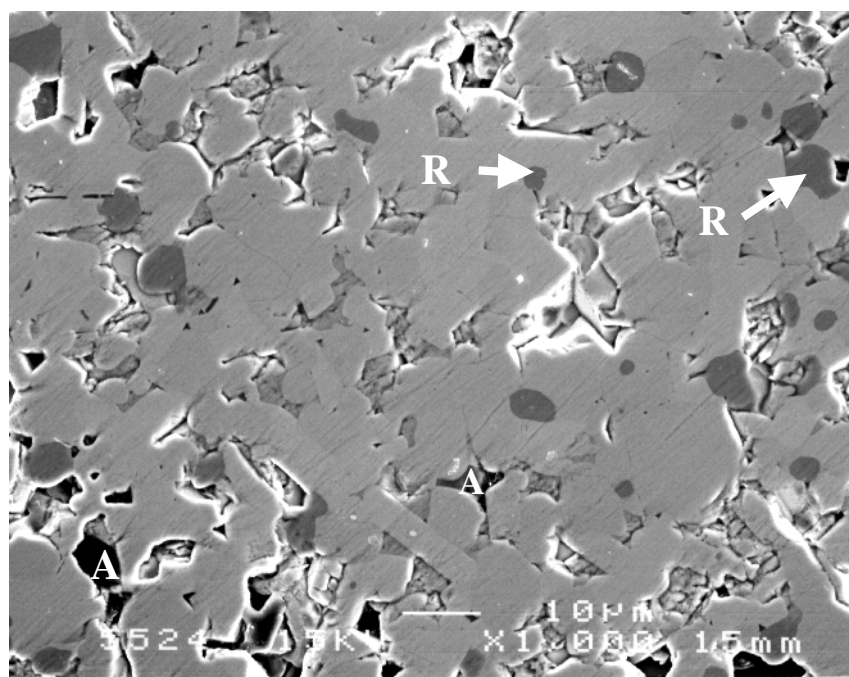
(a)



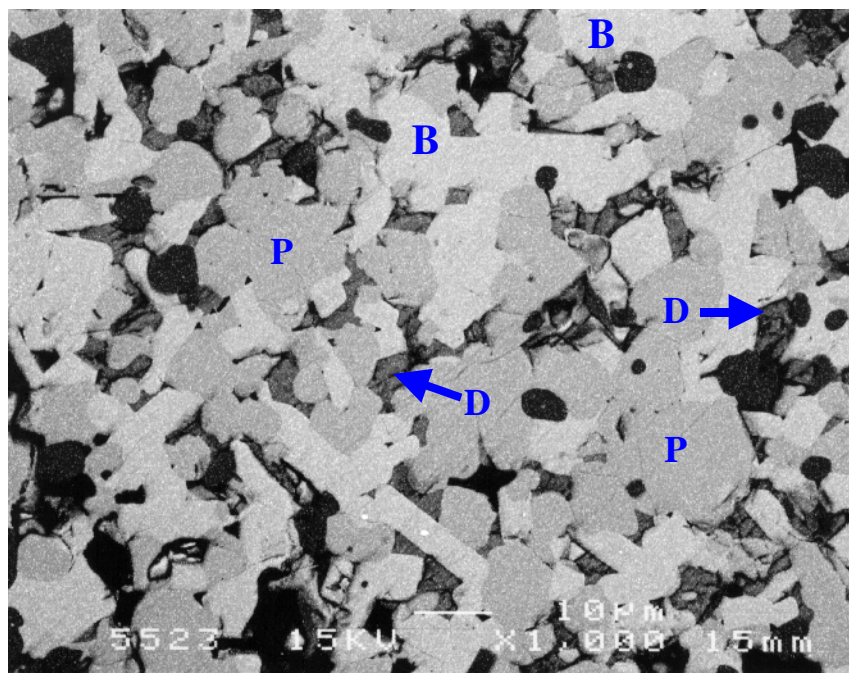
(b)

— 10 μm

Figure B-9: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu110-02 (Task 1.4, 5+ (Nb, Ta)-doped batch, sintered at 1350°C in air for 4 hours). The pellet consists of a pyrochlore matrix (P), brannerite (B, a lighter grey than pyrochlore), rutile (R, dark-grey grains), << 1 vol. % (Pu,U)O<sub>2</sub> (O, white spots associated with the brannerite), and porosity (A).



(a)



(b)

— 10 μm

Figure B-10: (a) Secondary electron micrograph and (b) backscattered electron micrograph of Pu111-02 (Task 1.4, 6+ (Mo, W)-doped batch, sintered at 1350°C in air for 4 hours). The pellet consists of a pyrochlore matrix (P), brannerite (B, light-grey grains), Hf-doped rutile (R, dark-grey grains), and powellite/scheelite ( $\text{Ca}(\text{Mo}, \text{W})\text{O}_4$ ) (D, mid-grey phase, with associated porosity) and porosity (A).

## **APPENDIX C**

**SCANNING ELECTRON MICROGRAPHS OF  
THE Pu/U-DOPED AND Ce/U-DOPED SAMPLES  
FIRED IN 3.7 % H<sub>2</sub>/ARGON AT 1350°C FOR 4  
HOURS**

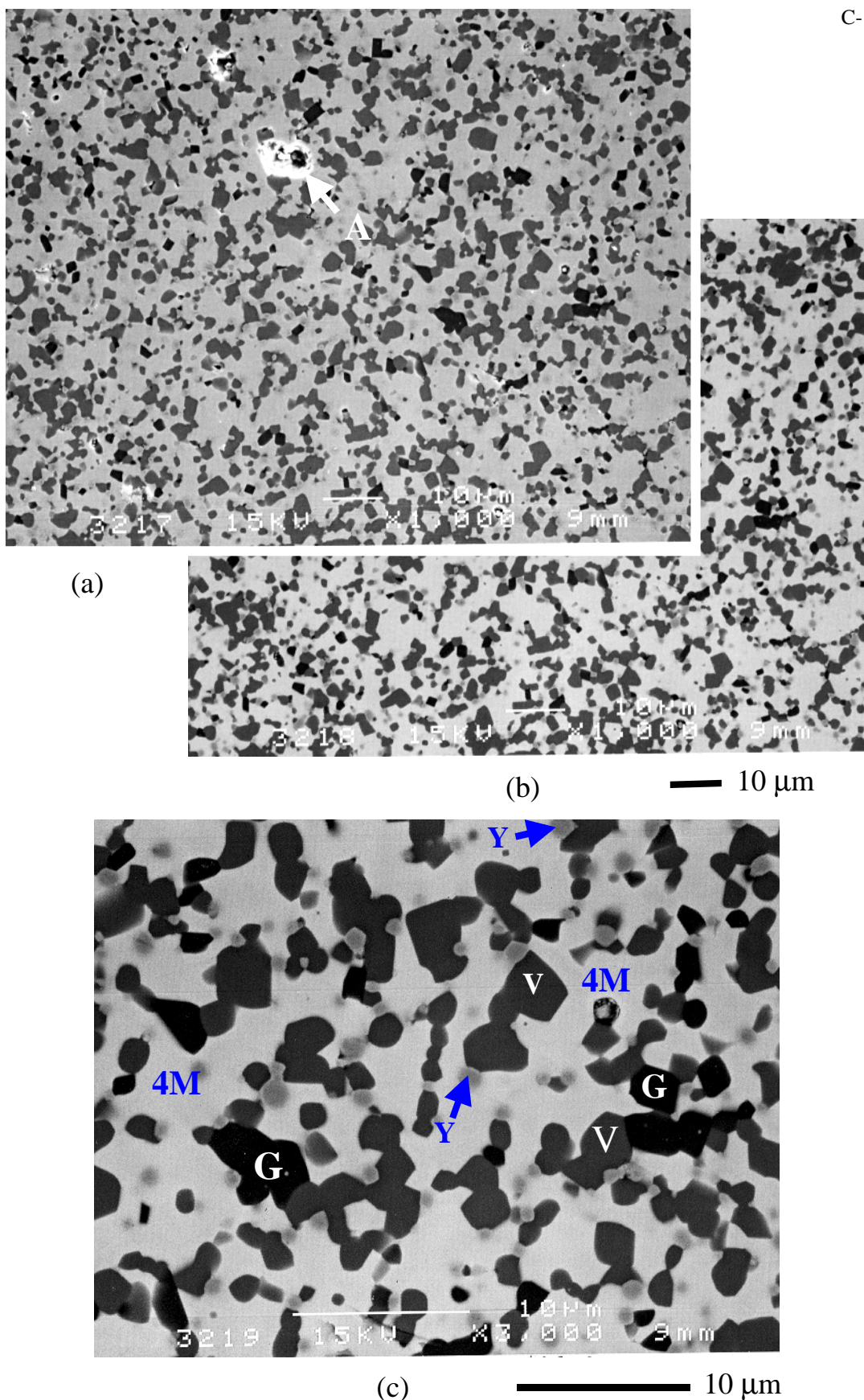
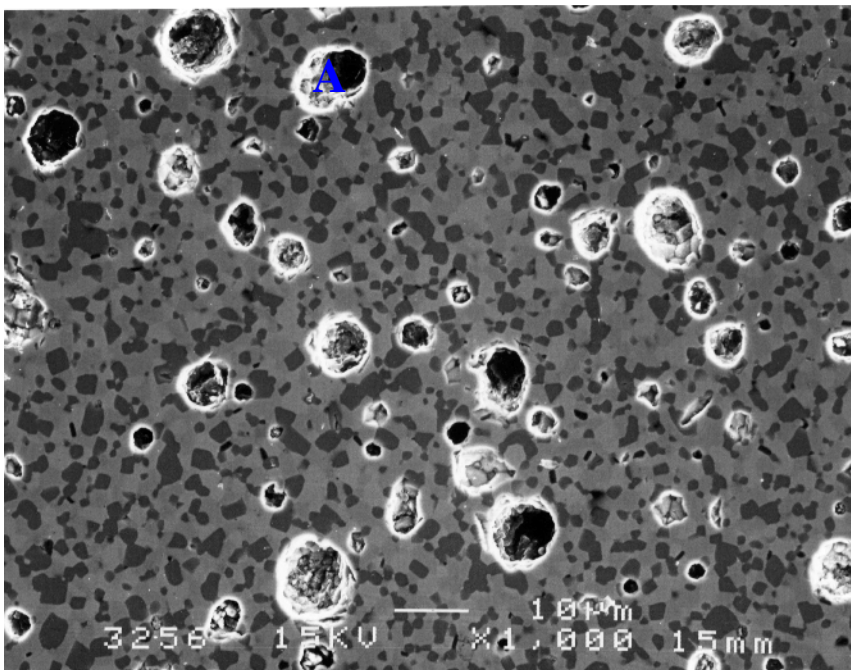
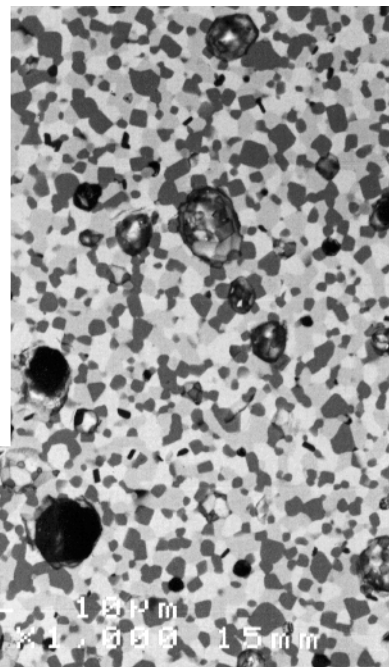


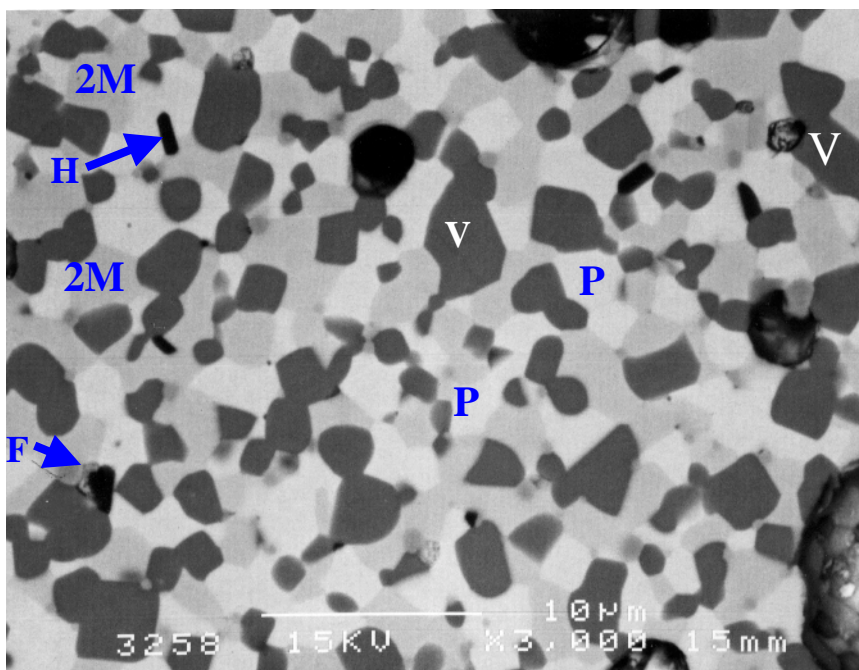
Figure C-1: (a) Secondary electron micrograph and, (b) and (c) backscattered electron micrograph of mws980336 (Task 1.4, 2+ (Mg, Ni, Cu, Zn, Fe, Mn)-doped batch, sintered at 1350°C in 3.7 % H<sub>2</sub>/Ar for 4 hours). The pellet consists of a matrix of what is believed to be 4M zirconolite (4M, light-grey), perovskite (V, darker grey), a Mg-Hf-Ti-O phase (G, dark-grey grains), metallic Fe-Cu-Ni-Co alloy (Y, small mid-grey grains) and a small



(a)

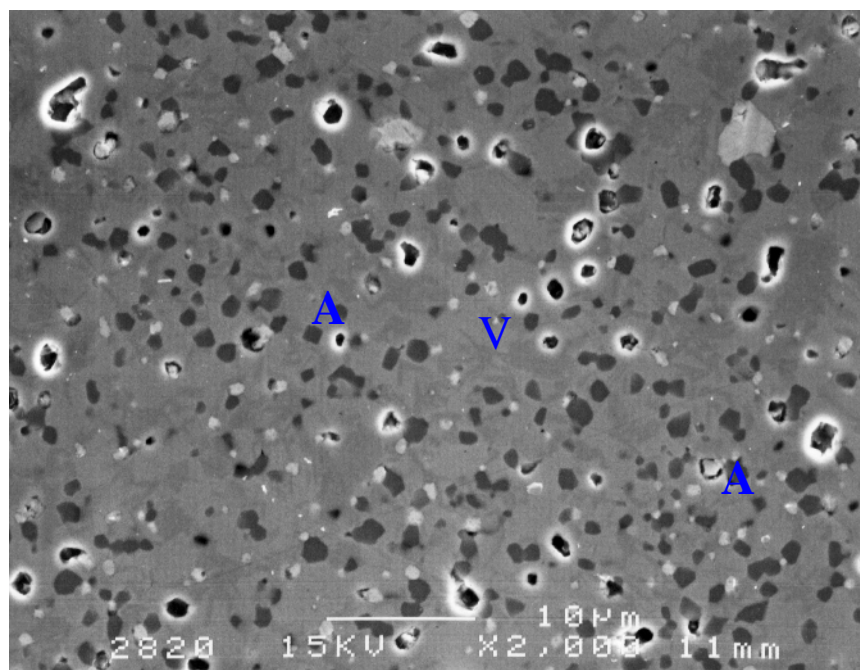


(b)

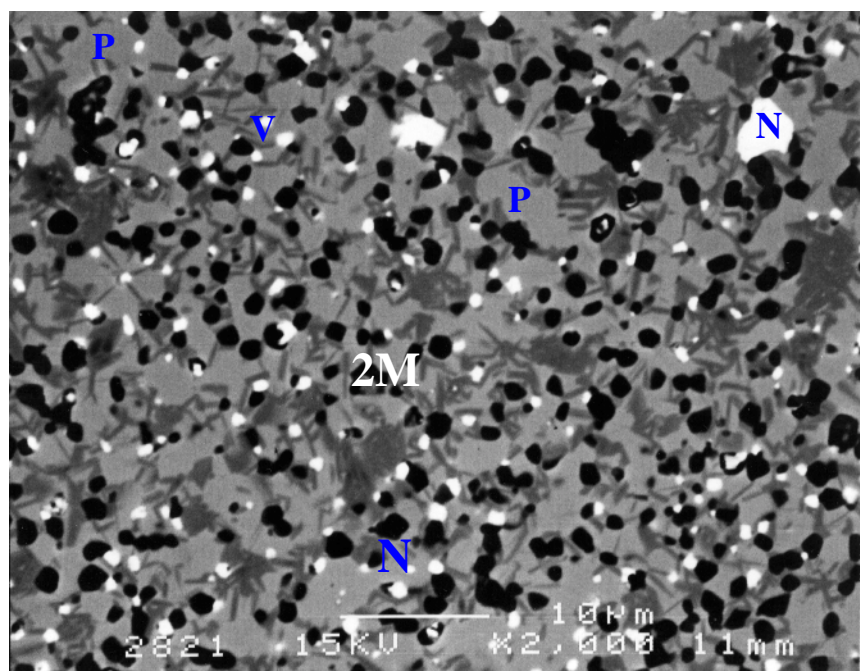


(c)

Figure C-2: (a) Secondary electron micrograph and, (b) and (c) backscattered electron micrograph of mws980337 (Task 1.4, 3+ (Al,Cr,Mn,Fe,Ga)-doped batch, sintered at 1350°C in 3.7 % H<sub>2</sub>/Ar for 4 hours). The pellet consists of pyrochlore (P, light-grey), 2M zirconolite (2M, light grey) perovskite (V, dark grey), Fe<sub>3</sub>Ga (F, small mid-grey grains), a small amount



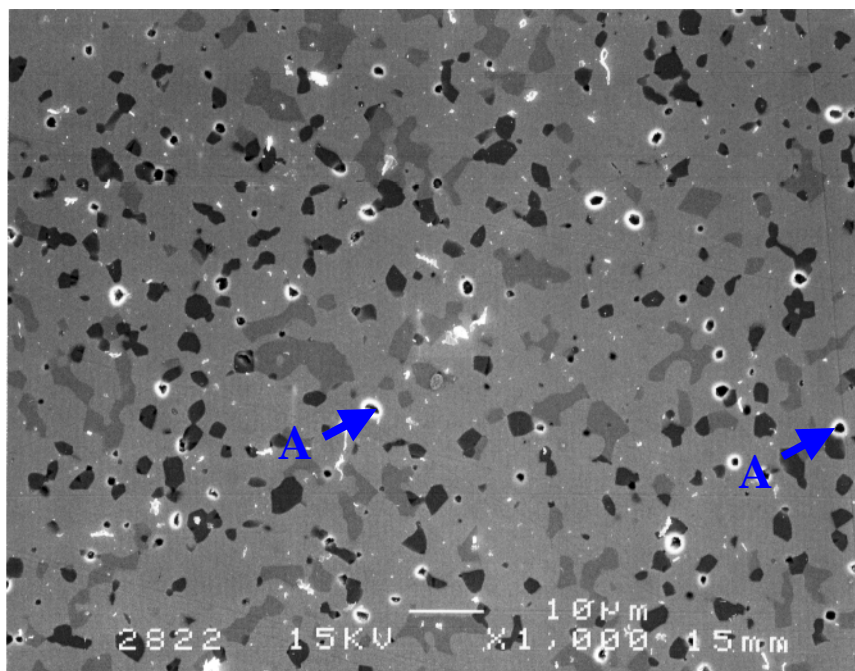
(a)



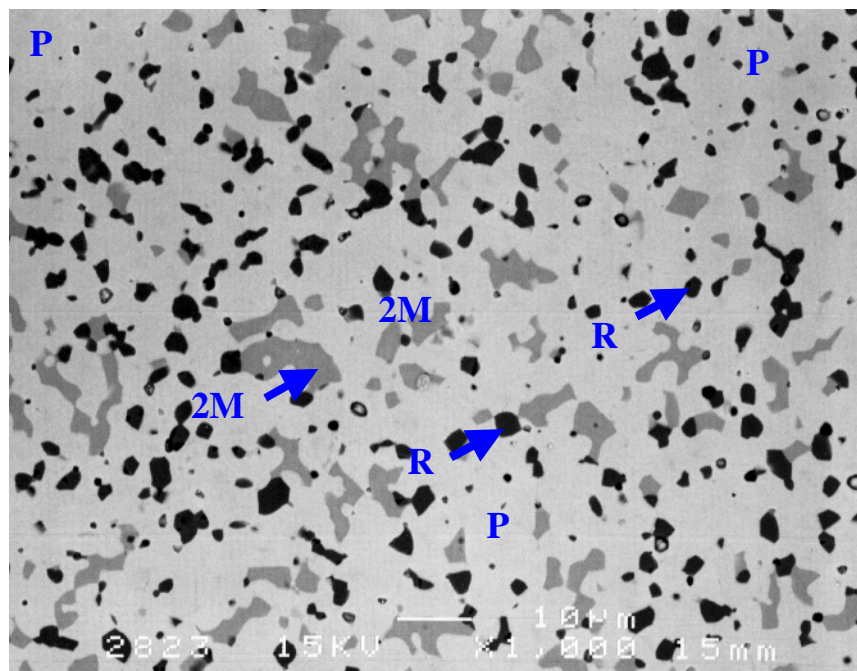
(b)

10 μm

Figure C-3: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980204 (Task 1.4, 4+ (Zr,Sn,Hf)-doped batch, sintered at 1350°C in 3.7 % H<sub>2</sub>/Ar for 4 hours). The pellet consists of pyrochlore (P, mid-grey), 2M zirconolite (2M, darker grey), Sn metal (N, light-grey-white grains), perovskite (V, dark-grey grains in (a)) and porosity (A).



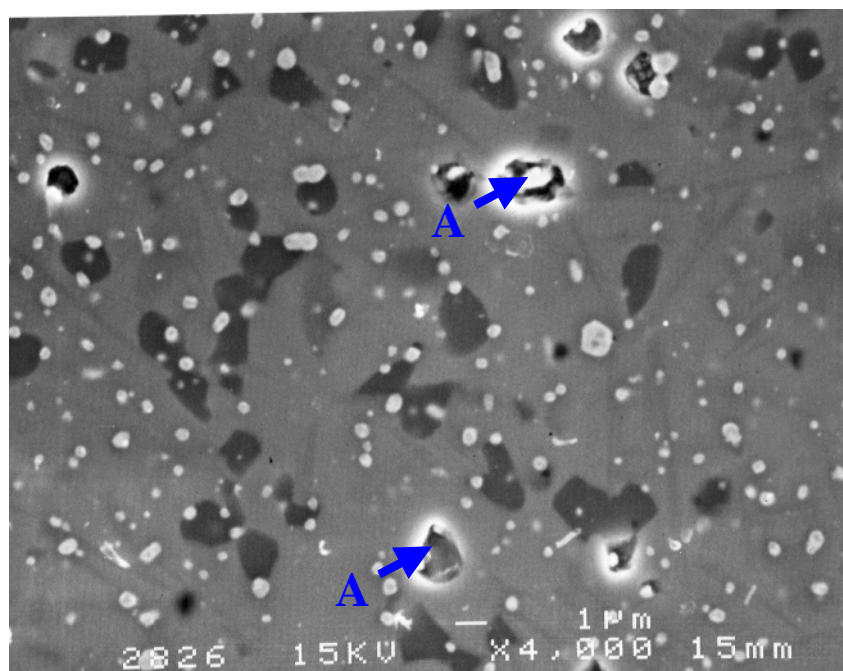
(a)



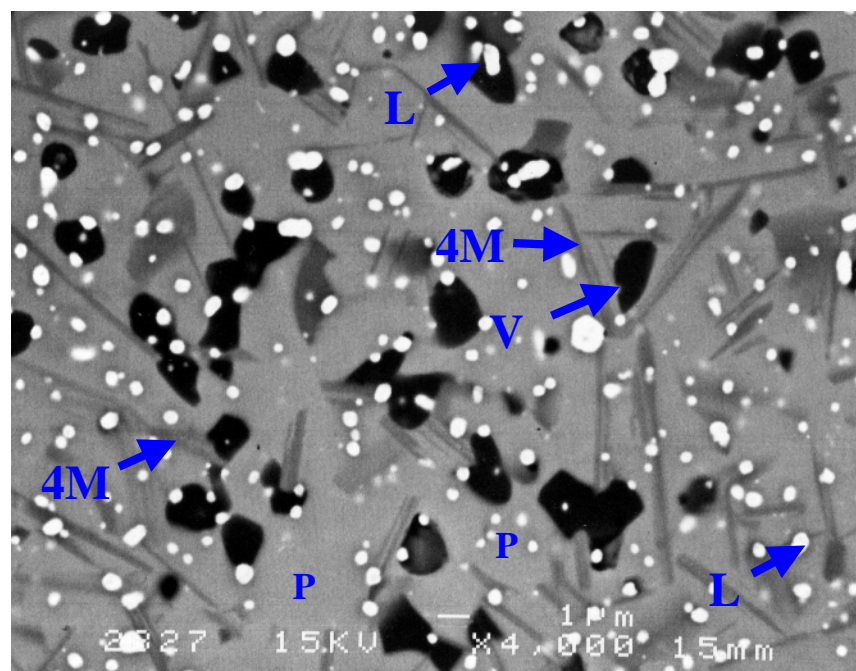
(b)

— 10 µm

Figure C-4: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980205 (Task 1.4, 5+ (Nb,Ta)-doped batch, sintered at 1350°C in 3.7 % H<sub>2</sub>/Ar for 4 hours). The pellet consists of a pyrochlore (P, light-grey) matrix with 2M zirconolite (2M, mid-grey), Ta-Nb-Hf-doped rutile (R, dark-grey grains) and porosity (A).



(a)



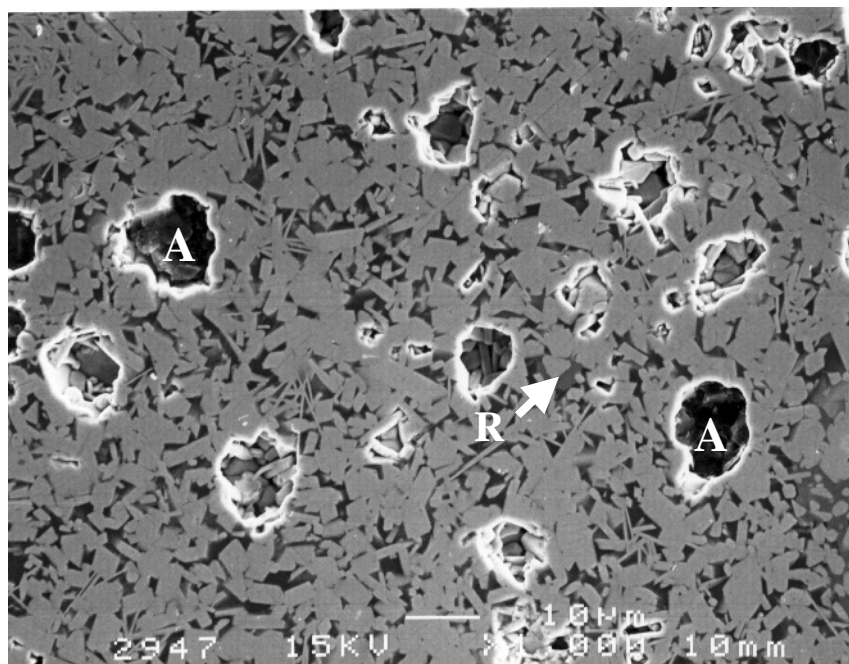
(b)

— 1 μm

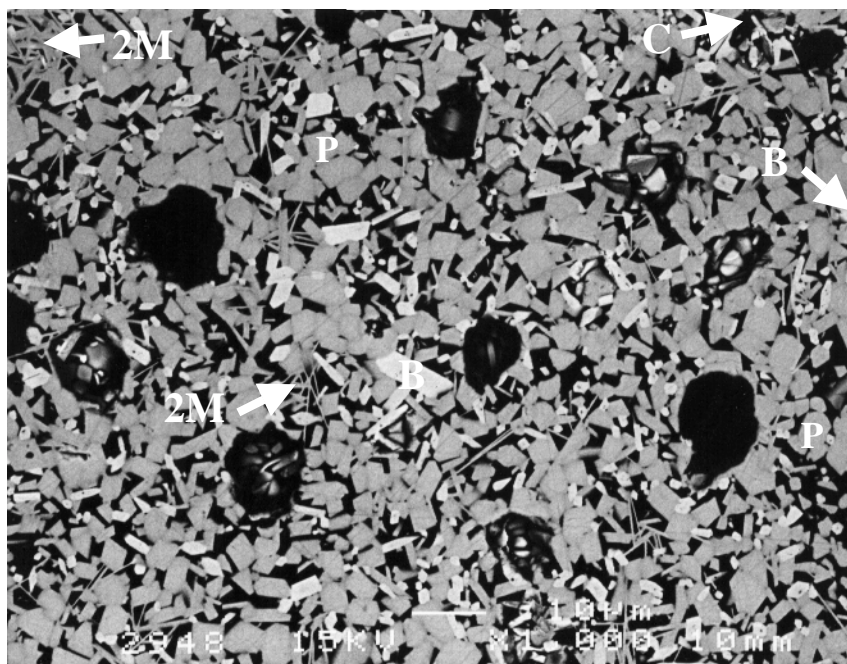
Figure C-5: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980206 (Task 1.4, 6+ (Mo,W)-doped batch, sintered at 1350°C in 3.7 % H<sub>2</sub>/Ar for 4 hours). The pellet consists of a pyrochlore (P) matrix with zirconolite, which is believed to be the 2M polytype (2M, needle-like phase), perovskite (V, dark-grey grains), a W-Mo metallic alloy (L, white spots) and porosity (A).

## **APPENDIX D**

### **SCANNING ELECTRON MICROGRAPHS OF THE LLNL GLASS-DOPED SAMPLES**



(a)

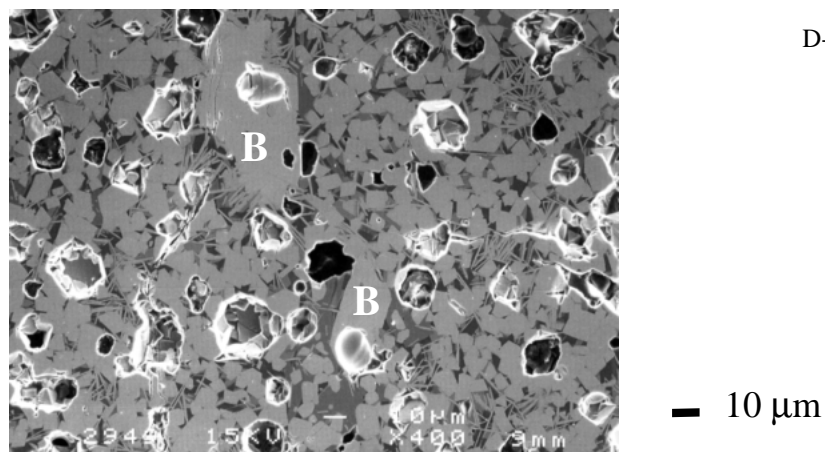


(b)

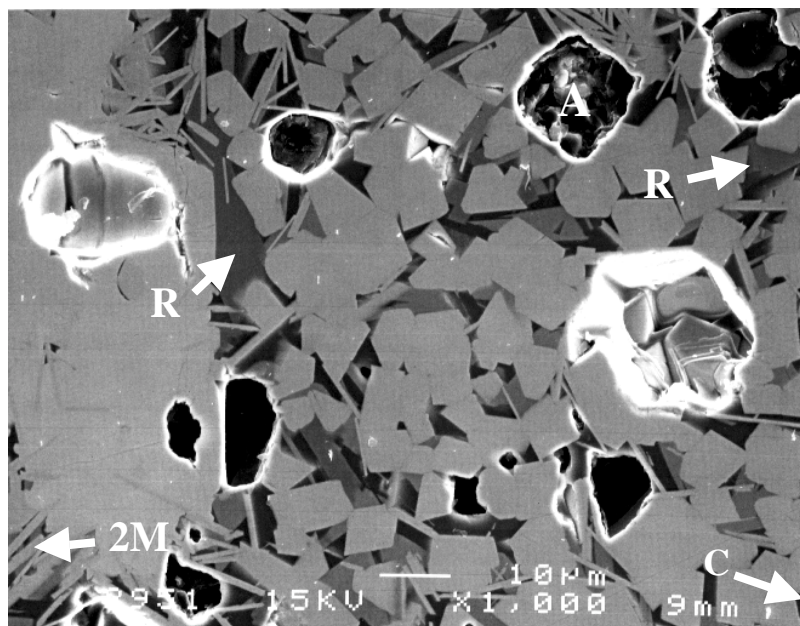
— 10 μm

Figure D-1: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980231 (Task 1.4, LLNL glass-doped batch, sintered at 1250°C in Ar for 4 hours). The pellet consists of pyrochlore (P, grey blocky grains), 2M zirconolite (2M, grey elongated grains), brannerite (B, whiteish grains), Hf-doped rutile (R, dark-grey phase), an intergranular silicate phase (black regions between the grains), a small amount of plagioclase (C, associated with the glass) and porosity (A).

(a)



(b)



(c)

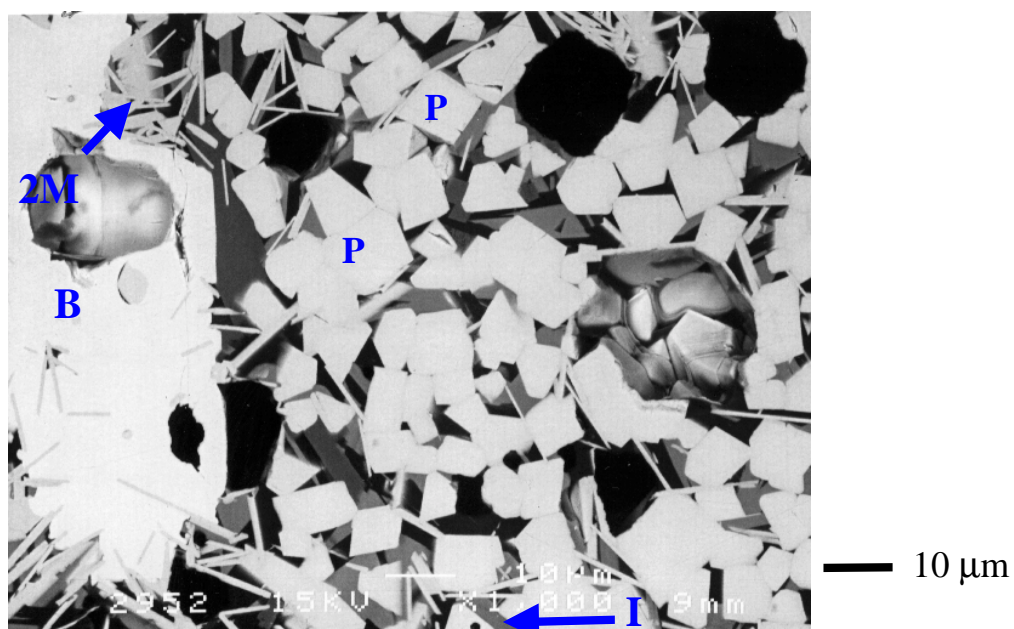
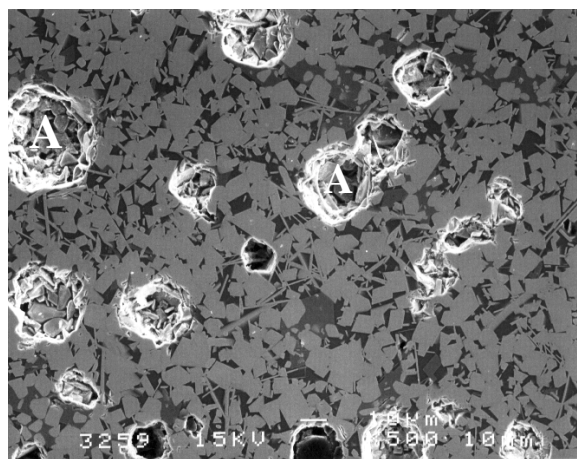
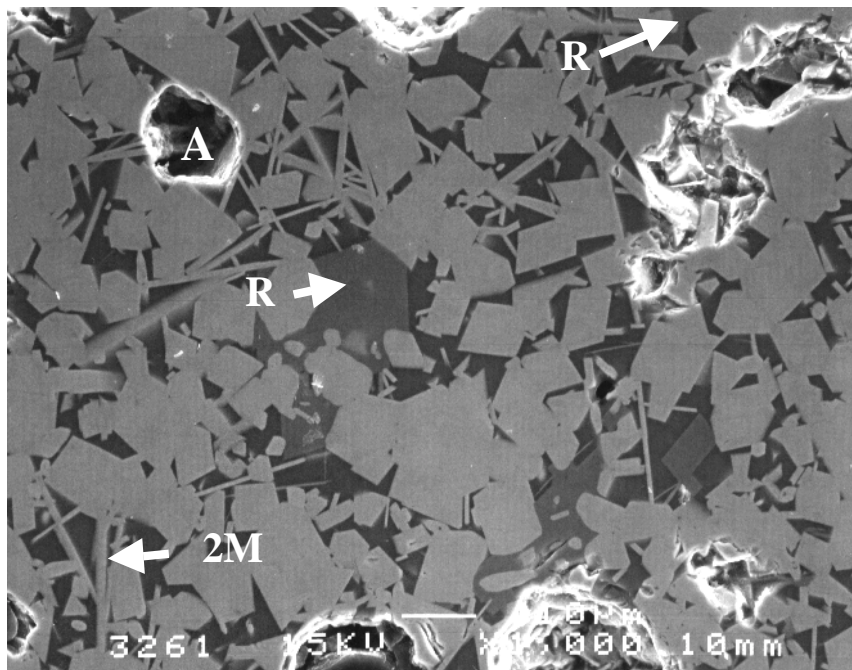


Figure D-2: (a) and (b) Secondary electron micrograph and (c) backscattered electron micrograph of mws980232 (Task 1.4, LLNL glass-doped batch, sintered at 1300°C in Ar for 4 hours). The pellet consists of pyrochlore (P. grey blocky grains), 2M zirconolite (2M. grey elongated grains), brannerite

(a)

— 10  $\mu\text{m}$ 

(b)



(c)

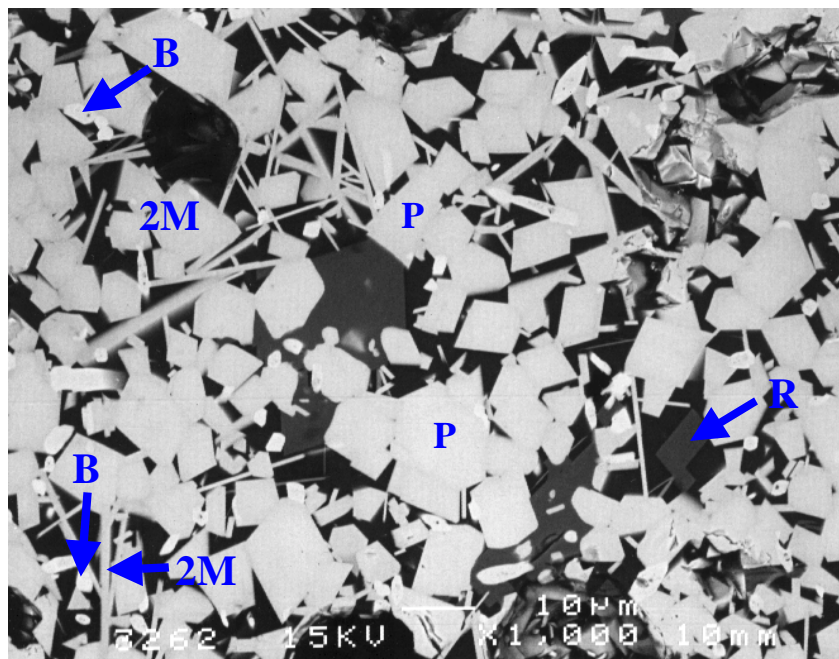
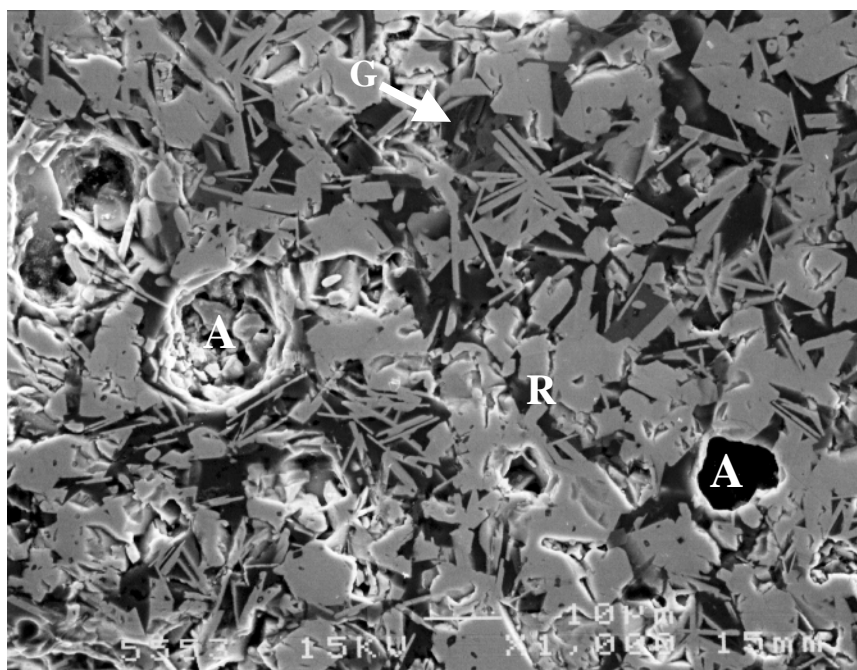
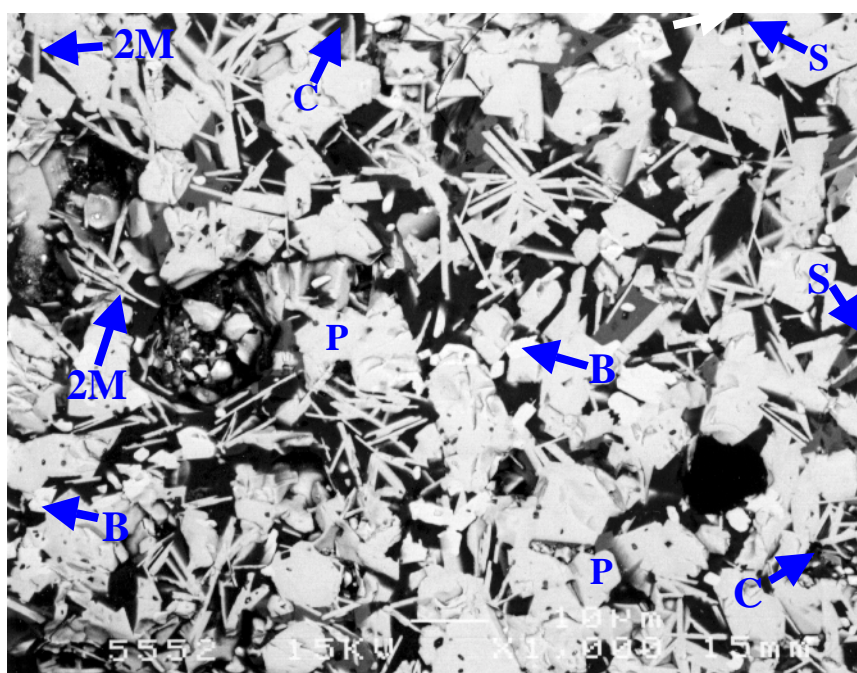
— 10  $\mu\text{m}$ 

Figure D-3: (a) and (b) Secondary electron micrographs and (c) backscattered electron micrograph of mws980338 (Task 1.4, LLNL glass-doped batch, sintered at 1350°C in Ar for 4 hours). The pellet consists of nvrochlore (P. grey blocky grains), 2M zirconolite (2M. grey elongated grains), brannerite



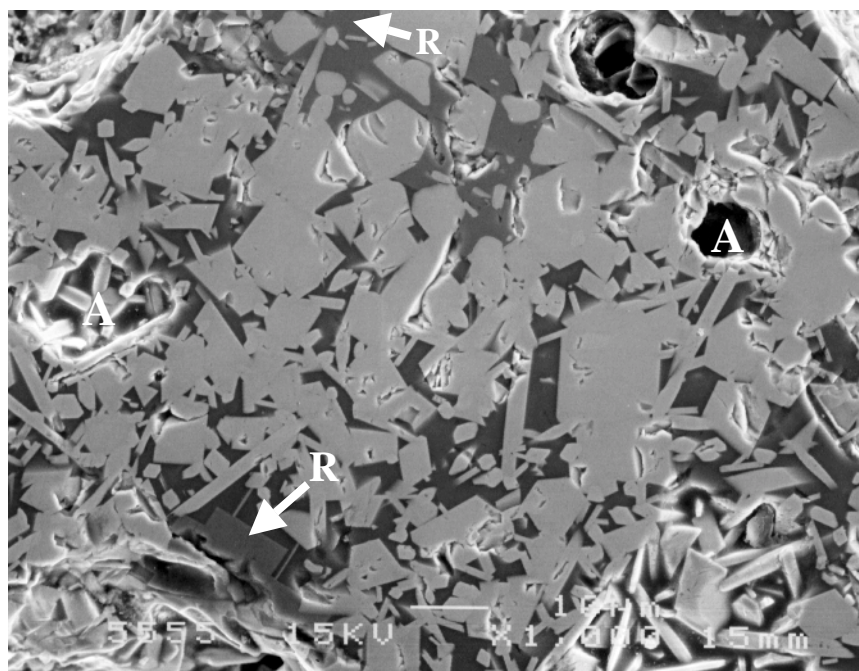
(a)



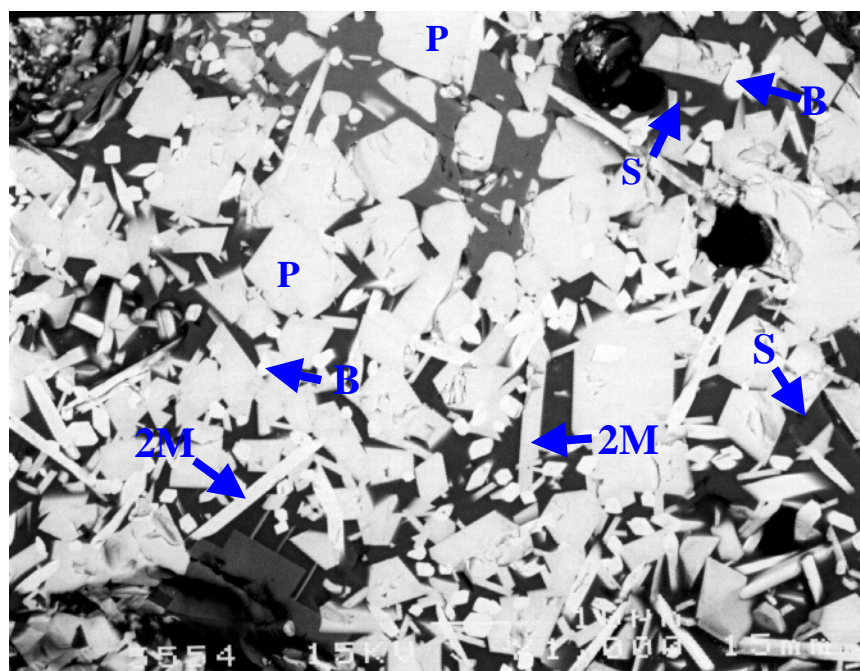
(b)

— 10  $\mu\text{m}$

Figure D-4: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980388 (Task 1.4, LLNL glass-doped batch with additional Al, sintered at 1300°C in Ar for 4 hours). The pellet consists of pyrochlore (P, light grey), 2M zirconolite (2M, grey elongated grains), brannerite (B, smaller whitish grains), Hf-doped rutile (R, dark-grey grains), magnetoplumbite (G), a small amount of silicate phase (S, black angular regions) and porosity (A).



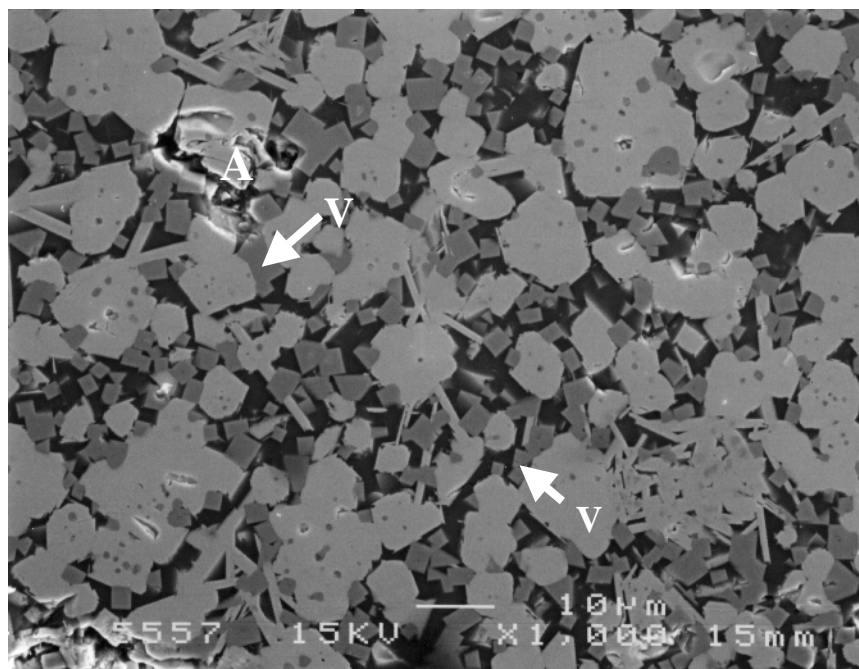
(a)



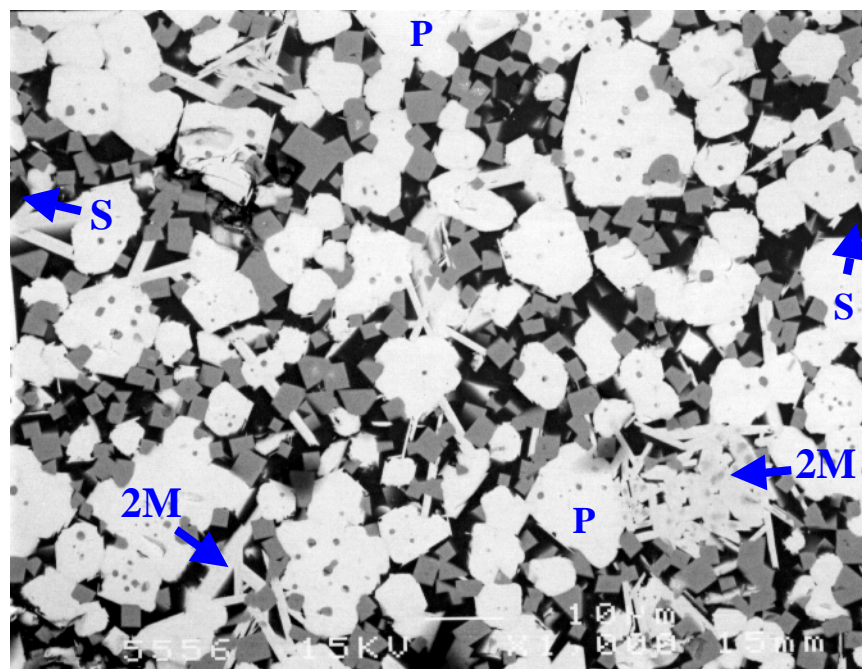
(b)

— 10 μm

Figure D-5: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980389 (Task 1.4, LLNL glass-doped batch with additional B, sintered at 1300°C in Ar for 4 hours). The pellet consists of pyrochlore (P, light grey), 2M zirconolite (2M, grey elongated grains), brannerite (B, smaller whitish grains), Hf-doped rutile (R, dark-grey grains), a silicate phase (S, dark grey regions between grains) and porosity (A).



(a)



(b)

— 10 μm

Figure D-6: (a) Secondary electron micrograph and (b) backscattered electron micrograph of mws980390 (Task 1.4, LLNL glass-doped batch with additional Na and K, sintered at 1300°C in Ar for 4 hours). The pellet consists of pyrochlore (P, light grey), 2M zirconolite (2M, grey elongated grains), perovskite (V, dark-grey phase), a silicate phase (S, dark grey-black regions between grains) and porosity (A).

## **APPENDIX E**

### **X-RAY DIFFRACTION RESULTS OF THE SAMPLES FROM TASK 1.4**

## E APPENDIX E X-RAY DIFFRACTION RESULTS

The following Table (E-1) contains a list of the raw data files. Note that those starting with t are Scintag Diffractometer files (Cu K-alpha radiation) and those starting with S are Siemens D500 Diffractometer data files (Co K-alpha). The patterns are from polished surfaces of pellets. The broad low angle peak in the patterns is from the resin used to mount the samples.

**Table E-1 Raw X-ray files of samples from the families of equivalent elements tested. Samples were sintered for 4 hours at 1350°C, except for the glass-doped batches.**

Description	Elements	Raw Data Files		
		Sintering atmosphere		
		Ar	Air	3.7% H <sub>2</sub> /Ar
<b><i>Ce/U-doped samples</i></b>				
2+	Mg, Co, Ni, Cu, Zn	t1446, S14397	-	-
2+ with Mn and Fe	Mg, Co, Ni, Cu, Zn, Mn, Fe	t1449, S14644	t1451, S14646	S14834
3+	Al, V, Cr, Mn, Fe, Ga	t1447, S14406	-	-
3+ no V	Al, Cr, Mn, Fe, Ga	t1450, S14645	t1452, S14647	S14641
4+	Sn, Zr, Hf	t1448, S14399	t1453, S14562	t1454, S14642
5+	Nb, Ta	S14400	S14563	S14642
6+	Mo, W	S14401	S14566	S14643
4+ actinides	Th, Np	t1240	-	-
<b><i>Pu/U-doped samples</i></b>				
2+ with Mn and Fe	Mg, Co, Ni, Cu, Zn, Mn, Fe	t1421	t1422	-
3+ no V	Al, Cr, Mn, Fe, Ga	t1419	t1423	-
4+	Sn, Zr, Hf	t1420	t1429	-
5+	Nb, Ta	t1345	t1425	-
6+	Mo, W	t1346	t1426	-
<b><i>Glass-doped</i></b>				
1250°C	Baseline + LLNL Glass	S15163		
1300°C	Baseline + LLNL Glass	S15162		
1350°C	Baseline + LLNL Glass	S14836, S15164		
1300°C	Baseline + LLNL Glass + Additional Al	S14944		
1300°C	Baseline + LLNL Glass + Additional B	S14945		
1300°C	Baseline + LLNL Glass + Additional K and Na	S14946		

The raw data files are on the enclosed computer disk.

Table E-2 contains the phases detected by XRD from the above patterns.

**Table E-2: Phases detected by x-ray diffraction**

Description	Elements	Phases Detected #,\$		
		Sintering atmosphere		
		Ar	Air	3.7% H <sub>2</sub> /Ar
<b><i>Ce/U-doped samples</i></b>				
2+	Mg, Co, Ni, Cu, Zn	Py, 2M, Pv, Us	-	-
2+ with Mn and Fe	Mg, Co, Ni, Cu, Zn, Mn, Fe	Py, 2M, Pv, Us	Py, 2M, Us	Py or 4M?, Pv, Fe
3+	Al, V, Cr, Mn, Fe, Ga	Py, 2M, L	-	-
3+ no V	Al, Cr, Mn, Fe, Ga	Py, 2M, Pv, L?	Py, 2M, L?	Py, 2M, Pv
4+	Sn, Zr, Hf	Py, 2M, 4M?, B, R	Py, B, R, H	Py or 4M?, 2M, Pv, Sn
5+	Nb, Ta	Py, B, R	Py, R	Py, 2M, R
6+	Mo, W	Py, B, R, Sc	Py, B, R, Sc	Py, Pv, R, Mo
4+ actinides	Th, Np	Py, B, A	-	-
<b><i>Pu/U-doped samples</i></b>				
2+ with Mn and Fe	Mg, Co, Ni, Cu, Zn, Mn, Fe	Py, Us	Py, 2M, Us	
3+ no V	Al, Cr, Mn, Fe, Ga	Py, 2M, L?	Py, 2M, L	
4+	Sn, Zr, Hf	Py, 2M, B, H?	Py, B, H	
5+	Nb, Ta	Py, B, R	Py, B, R	
6+	Mo, W	Py, B, R, Sc	Py, B, R, Sc	
<b><i>Glass-doped</i></b>				
1250°C	Baseline + LLNL Glass	Py, B, R, 2M	-	-
1300°C	Baseline + LLNL Glass	Py, B, R, 2M?	-	-
1350°C	Baseline + LLNL Glass	Py, B, R	-	-
1300°C	Baseline + LLNL Glass + Additional Al	Py, B, 2M R?	-	-
1300°C	Baseline + LLNL Glass + Additional B	Py, B, 2M?, R?	-	-
1300°C	Baseline + LLNL Glass + Additional K and Na	Py, Pv, 2M?	-	-

# Py = pyrochlore, 4M = 4M zirconolite, 2M = 2M zirconolite, H = Hf-Zr titanate, R = hafnia-doped rutile, B = brannerite (Ce/U-brannerite, Th/U-brannerite or Pu/U-brannerite), Pv = Perovskite, Us = ulvospinel, L = loveringite, Sc = scheelite-powellite (Ca(Mo,W)O<sub>4</sub>) solid solution, A = actinide-rare earth oxide, Sn = metallic tin, Mo = W-Mo alloy, Fe = Fe-Ni-Co-Cu alloys

\$ ? = not yet rigorously identified, either due to small peaks and/or possible overlap with major peaks from other phases, or issue still to be resolved (e.g., 4M vs pyrochlore)

## **APPENDIX F**

### **ENERGY DISPERSIVE X-RAY SPECTROSCOPY RESULTS ON THE SCANNING ELECTRON MICROSCOPY SAMPLES SHOWN IN APPENDICES A TO D**

## APPENDIX F: EDS ANALYSES OF PHASES IN TASKS 1.4

### 1.1 KEY TO PHASE ABBREVIATIONS USED IN TABLES

oxide	(U,Pu)O <sub>2</sub>
bran	brannerite
pyr	pyrochlore
2M zirc	2M zirconolite
4M zirc	4M zirconolite
per	perovskite
rutile	rutile
ulvo	ulvospinel
Mg-Ti-O	unidentified magnesium titanate phase
lovng	lovingite-like phase
mplm	magnetoplumbite
plagio	plagioclase
alloy	metallic alloy
silicate	alumino-silicate phase, probably a glass